# Handbook of Methods for Acid Deposition Studies Field Operations for Surface Water Chemistry

A Contribution to the National Acid Precipitation Assessment Program

U. S. Environmental Protection Agency
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#### Notice

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Mention of corporation names, trade names, or commercial products does not constitute endorsement or recommendation for use.

This document is a contribution to the National Acid Precipitation Assessment Program. The methods described in this document have been developed for use in the component programs of the Aquatic Effects Research Program. Previous publications from which these methods have been extracted include:

- Bonoff, M. B., and A. W. Groeger, 1987. National Surface Water Survey, Western Lake Survey Phase I (Synoptic Chemistry) Field Operations Report. EPA 600/8-87/018. U.S. Environmental Protection Agency, Environmental Monitoring Systems Laboratory, Las Vegas, Nevada.
- Hagley, C. A., C. L. Mayer, and R. Hoenicke. 1988. National Stream Survey Phase I Field Operations Report. EPA 600/4-88/023. U.S. Environmental Protection Agency, Las Vegas, Nevada. 36 pp.
- Knapp, C. M., C. L. Mayer, D. V. Peck, J. R. Baker, and G. J. Filbin. 1987. National Surface Water Survey: National Stream Survey - Phase I Pilot Survey Field Operations Report. EPA 600/8-87/019. U.S. Environmental Protection Agency, Environmental Monitoring Systems Laboratory, Las Vegas, Nevada.
- Morris, F. A., D. V. Peck, M. B. Bonoff, K. J. Cabble, and S. L. Pierett. 1986. National Surface Water Survey, Eastern Lake Survey (Phase I Synoptic Chemistry) Field Operations Report. EPA 600/4-86/010, U.S. Environmental Protection Agency, Las Vegas, Nevada. 46 pp.

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#### Abstract .

This handbook describes methods used to collect surface water samples of low ionic strength. It is intended as a guidance document for groups involved in acidic deposition monitoring activities similar to those implemented by the Aquatic Effects Research Program.

The handbook defines the logistical needs of field operations. These concerns include designing a survey, establishing base sites and sampling schedules, defining field personnel needs, developing sampling protocols, and implementing pilot surveys, communications networks, and training programs. The handbook also describes lake and stream sampling operations, describes various means of access, and provides standard operating procedures for physical and chemical measurements.

The methods decribed in the handbook were developed for use in component projects of the Aquatic Effects Research Program under the Acid Deposition and Atmospheric Research Division of the Office of Acid Deposition, Environmental Monitoring, and Quality Assurance, U.S. Environmental Protection Agency. This program addresses the following questions relating to the effects of acidic deposition on aquatic ecosystems:

- 1. The extent and magnitude of past change.
- 2. The change to be expected in the future under various deposition scenarios.
- 3. The maximum rates of deposition below which further change is not expected.
- 4. The rate of change or recovery of aquatic ecosystems if deposition rates are decreased.

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### Acronyms, Abbreviations, and Symbols

#### Acronyms

#### **AERP** Aquatic Effects Research Program ANC acid neutralizing capacity **ASTM** American Society of Testing and Materials **ATC** automatic thermo compensator BOD biological oxygen demand **BRC** Biologically Relevant Chemistry project **CPR** cardiopulmonary resuscitation **DDRP** Direct/Delayed Response Project DIC dissolved inorganic carbon DO dissolved oxygen Eastern Lake Survey **ELS** ELS-I Eastern Lake Survey - Phase I ELS-II Eastern Lake Survey - Phase II ELT **Emergency locator transmitter EPA** U.S. Environmental Protection Agency **ERP Episodic Response Project** FAA Federal Aviation Administration ID identification IHHE Indirect Human Health Effects project LCD liquid crystal display **MIBK** methyl isobutyl ketone NAPAP National Acid Precipitation Assessment Program **NBS** National Bureau of Standards **NLS** National Lake Survey NSS **National Stream Survey** NSWS National Surface Water Survey OAS Office of Aircraft Services **PCV** pyrocatechol violet QA/QC quality assurance and quality control QCC quality control check **REW** right edge of the water **SBRP** Southern Blue Ridge Province SRM standard reference material TIME Temporally Integrated Monitoring of Ecosystems project **USGS** United States Geological Survey VDC volts direct current WLS Western Lake Survey YSI Yellow Springs Instrument Company

#### Abbreviations and Symbols

°C	degrees centrigrade
cm	centimeter
CO2	carbon dioxide
ft	feet
g	gram
gal	gallon
Ĥ <sup>+</sup>	hydrogen ion
H <sub>2</sub> SO <sub>4</sub>	sulfuric acid
HgCl <sub>2</sub>	mercuric chloride
KČI -	potassium chloride
L	liter
M	Molar
m ·	meter
mg/L	milligrams per liter
mL	milliliter
mm	millimeter
m/sec	meters per second
N	normality
NaOH	sodium hydroxide
ppm	parts per million
psi	pounds per square inch
qt	quart
W/V	weight to volume ratio
μmhos/cm	
μS/cm	micro Siemans per centimeter
%	percent
±	plus or minus

### Acknowledgments

In addition to the publications listed in the Notice, methods presented in this handbook are based on methods contained in the following internal reports:

- Bonoff, M. B., K. J. Cabble, D. J. Chaloud, and L. A. Drewes. 1986. National Surface Water Survey, Eastern Lake Survey (Phase II Spring Variability Study, Pilot) Training Manual. U.S. Environmental Protection Agency, Las Vegas, Nevada. Internal Report. 139 pp.
- Cabble, K. J., and G. D. Merritt. 1986. National Surface Water Survey, Eastern Lake Survey (Phase II Spring Variability Pilot Study and Spring Overturn Survey) Field Personnel Training Report. U.S. Environmental Protection Agency, Las Vegas, Nevada, Internal Report. 14 pp.
- Drewes, L. A., K. J. Cabble, D. J. Chaloud, A. W. Groeger, and M. B. Bonoff. 1936. National Surface Water Survey, Eastern Lake Survey (Phase II Temporal Variability) Field Operations Manual for Summer Sampling. U.S. Environmental Protection Agency, Las Vegas, Nevada. Internal Report. 87 pp.
- Groeger, A. W., D. J. Chaloud, and M. B. Bonoff. 1986. National Surface Water Survey, Eastern Lake Survey (Phase II Temporal Variability and Biological Resources) Field Operations Manual for Spring, Summer, and Fall Sampling. U.S. Environmental Protection Agency, Las Vegas, Nevada. Internal Report. 49 pp.
- Hagley, C. A. 1986. National Surface Water Survey National Stream Survey Summary of Training Activities. U.S. Environmental Protection Agency, Las Vegas, Nevada. Internal Report. 19 pp.
- Hagley, C. A., C. M. Knapp, C. L. Mayer, and F. A. Morris. 1986. National Surface Water Survey, National Stream Survey (Middle-Atlantic Phase I, Southeast Screening, and Middle-Atlantic Episodes Pilot) Field Training and Operations Manual. U.S. Environmental Protection Agency, Las Vegas, Nevada. Internal Report. 126 pp.
- Hillman, D. C., D. V. Peck, J. R. Baker, F. A. Morris, K. J. Cabble, and S. L. Pierett. 1985. National Stream Survey Pilot Study Field Training and Operations Manual. U.S. Environmental Protection Agency, Las Vegas, Nevada. Internal Report. 158 pp.
- Merritt, G. D. 1986. National Surface Water Survey Eastern Lake Survey (Phase II Summer Stratification Survey) Training Report. U.S. Environmental Protection Agency, Las Vegas, Nevada. Internal Report. 8 pp.
- Metcalf, R. C., J. R. Wilson, G. D. Merritt, and M. E. Mitch. 1986. National Surface Water Survey, Eastern Lake Survey (Phase II 1986 Spring Variability Pilot Survey) Field Operations Report. U.S. Environmental Protection Agency, Las Vegas, Nevada. Internal Report. 39 pp.
- Morris, F. A., L. A. Drewes, and D. V. Peck. 1986. National Surface Water Survey, Western Lake Survey (Phase I) Field Personnel Training Report. U.S. Environmental Protection Agency, Las Vegas, Nevada. Internal Report. 60 pp.

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- Morris, F. A., D. C. Hillman, R. F. Cusimano, K. J. Cabble, S. L. Pierett, and W. L. Kinney. 1985.
  National Surface Water Survey, Phase IA1 Field Training and Operations Manual. U.S.
  Environmental Protection Agency, Las Vegas, Nevada. Internal Report. 178 pp.
- Morris, F. A., D. V. Peck, D. C. Hillman, K. J. Cabble, S. L. Pierett, and W. L. Kinney. 1985. National Surface Water Survey, Western Lake Survey (Phase I) Field Training and Operations Manual. U.S. Environmental Protection Agency, Las Vegas, Nevada. Internal Report. 201 pp.
- Nicholson, J. M., and V. A. Sheppe. 1986. National Surface Water Survey, Eastern Lake Survey (Phase II) Fall Chemistry Survey Training Report. U.S. Environmental Protection Agency, Las Vegas, Nevada. Internal Report. 8 pp.
- Peck, D. V., and C. M. Knapp. 1985. National Stream Survey Pilot Study, Summary of Training Activities. U.S. Environmental Protection Agency, Las Vegas, Nevada. Internal Report. 18 pp.
- Peck, D. V., R. F. Cusimano, and W. L. Kinney. 1985. National Surface Water Survey, Western Lake Survey (Phase I Synoptic Chemistry) Ground Samplers Training and Operations Manual. U.S. Environmental Protection Agency, Las Vegas, Nevada. Internal Report. 44 pp.
- Todechiney, L. R., K. J. Cabble, and J. R. Wilson. 1986. National Surface Water Survey, Eastern Lake Survey (Phase II Temporal Variability) Field Operations Manual for Fall Sampling. U.S. Environmental Protection Agency, Las Vegas, Nevada. Internal Report. 87 pp.

### 1.0 Introduction to the Aquatic Effects Research Program

#### 1.1 Overview

Concern over the effects of acidic deposition on the nation's surface water resources led the U.S. Environmental Protection Agency (EPA) to initiate research in the field in the late 1970's. Early research, focusing on a diversity of potential effects, provided insight into those research areas which were considered central to key policy questions. Recognizing the need for an integrated, stepwise approach to resolve the issues, EPA implemented the Aquatic Effects Research Program (AERP) in 1983 with its present structure, focus, and approach. The program, a part of the EPA Office of Research and Development, is administered by the Acid Deposition and Atmospheric Research Division in the Office of Acid Deposition, Environmental Monitoring, and Quality Assurance. The AERP is also a major component of the National Acid Precipitation Assessment Program's (NAPAP) Aquatic Effects Research Task Group 6, a cooperative effort of nine federal agencies tasked with addressing important policy and assessment questions relating to the acidic deposition phenomenon and its effects.

This handbook of methods for field operations related to determining water chemistry is an outgrowth of several AERP surveys. The purpose of this handbook is to provide general guidelines and procedures, derived from specific AERP surveys, that can be adapted readily by different research groups involved in acidic deposition monitoring activities.

Initially, AERP studies focused on process-oriented research at a few sites to generate hypotheses for further testing and to identify key parameters associated with the effects of acidic deposition on aquatic ecosystems. In 1983, after it was determined that regional assessments of the effects of acidic deposition could not be made with confidence on the basis of available historical data, the AERP redirected its focus to provide the required information. Weaknesses of available data included possible inconsistencies in the selection of study sites, lack of data for certain important parameters, inconsistent sampling and analytical methods, and little or no information on quality assurance.

The AERP addresses four major policy questions relating to the effects of acidic deposition on aquatic ecosystems:

- The extent and magnitude of past change.
- 2. The change to be expected in the future under various deposition scenarios.
- 3. The maximum rates of deposition below which further change is not expected.
- 4. The rate of change or recovery of aquatic ecosystems if deposition rates are decreased.

An integrated, stepwise approach has been designed within the AERP to provide the necessary data for assessment and policy decisions related to effects of acidic deposition on aquatic resources. The approach employs statistically based site selection, standardized sampling procedures and analytical methods, and rigorous quality assurance protocols. The AERP includes five major research projects: the National Surface Water Survey (NSWS), the Direct/Delayed

Response Project (DDRP), the Episodic Response Project (ERP), the Watershed Processes and Manipulations Project (WMP), and the Temporally Integrated Monitoring of Ecosystems (TIME) Project. Two additional projects, Biologically Relevant Chemistry (BRC) Project and Indirect Human Health Effects (IHHE), have been incorporated into the AERP research design. The AERP projects form an integrated program to quantify the chemical status of surface waters, to predict the response of biologically relevant water chemistry to variable rates of acidic deposition, and to verify and validate the predictions.

The AERP projects are concerned primarily with assessing chronic, or long-term, acidification of surface waters which are affected by sulfur deposition. The Episodic Response Project assesses the importance of acute, or short-term, acidification and nitrate deposition. Components of the Biologically Relevant Chemistry Project address issues of both chronic and acute acidification.

### 1.2 National Surface Water Survey (NSWS)

The NSWS is divided into two components: the National Lake Survey (NLS) and the National Stream Survey (NSS). Figure 1-1 shows the various regions sampled during the NSWS.

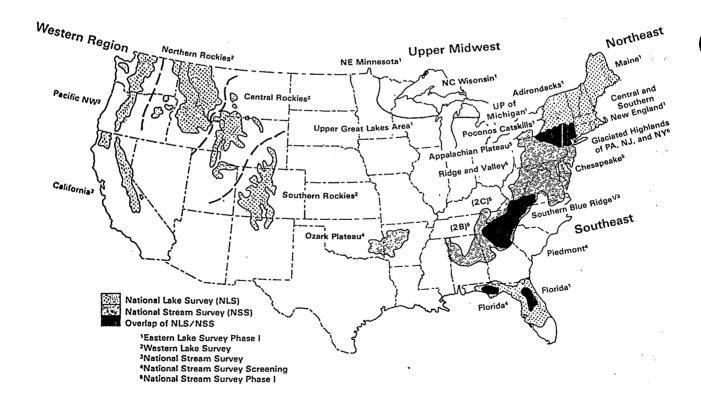


Figure 1-1. Regions sampled during the National Surface Water Survey.

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The initial phase of the NLS consisted of the Eastern Lake Survey (ELS) and the Western Lake Survey (WLS). The surveys, conducted in 1984 in the northeastern, midwestern, and southeastern United States (ELS) and in 1985 in mountainous areas of the western United States (WLS), provided baseline information about the current chemical status of our nation's lakes. A single water sample was collected from each NLS lake during fall turnover, a period of minimum within-lake chemical variability. This index sample represented the integration of chemical inputs and lake transformation processes which occur during other seasons.

During ELS, scientists used helicopters to access and sample 1,798 lakes (with surface areas between 4 and 2,000 hectares). Samples from 757 lakes (with surface areas between 1 and 2,000 hectares) were collected during WLS. Approximately one-half of the WLS lakes were located within designated wilderness areas. U.S. Department of Agriculture Forest Service personnel reached these lakes by foot or pack animal.

The second phase of NLS was initiated in the northeastern United States in 1986 and included three seasonal chemistry surveys. Each of 147 lakes, selected from lakes sampled during Phase I of the ELS, was sampled during the spring, summer, and fall at approximately the same location on the lake sampled during Phase I. These surveys provided data necessary to characterize seasonal patterns in water chemistry and to relate these patterns to the fall index conditions of Phase I.

The NSS was conducted in the Southern Blue Ridge Province (SBRP), the Mid-Atlantic states, and the Southeast. Designed to provide baseline chemistry information about streams, the NSS included three components: a feasibility study (1985), the Mid-Atlantic Survey (1986), and the Southeastern Screening Survey (1986).

## 1.3 Direct/Delayed Response Project (DDRP)

The purpose of DDRP is to provide data on watersheds and soils to complement the surface water data of the NSWS. These data are used in three watershed acidification models to predict the time scales over which surface waters are expected to become chronically acidic, given different levels of acidic inputs.

The northeastern United States and the SBRP were studied during initial DDRP activities. Sampling at 180 sites in these two regions was completed by the winter of 1986. An additional 37 watersheds were sampled in the fall of 1988 in the Mid-Appalachian region.

### 1.4 Episodic Response Project (ERP)

The ERP has objectives similar to those of NSWS, but focuses on the magnitude, frequency, and duration of episodic acidification and the effect of episodes on regional water chemistry and watershed processes. The ERP is being conducted at a small number of watersheds believed to represent the range of conditions found within a region, based on the results of NSWS and DDRP. Empirical and conceptual models are being developed from these site-specific studies to address the regional extent of episodes, using the NSWS statistical frame. The Fernow Experimental Forest, a U.S. Department of Agriculture Forest Service research site, is the site for the first ERP experimental studies.

### 1.5 Watershed Processes and Manipulations

The Watershed Processes and Manipulations project, involving process-oriented research at a small number of watersheds, is designed to assess the quantitative and qualitative response of watershed soils and surface waters to altered deposition. Data gathered during this project provides information about the interactions among biogeochemical mechanisms controlling the response of surface waters to acidic inputs at various scales within watersheds, ranging from plot to whole ecosystem studies. A number of complimentary studies, currently ongoing at a number of sites, are included in this research effort. These studies include the Little Rock Lake Experimental Acidification Project (northern Wisconsin), the Watershed Manipulation Project (southeastern Maine), and the Regional Episodic and Acidic Manipulation Project (Fernow, West Virginia).

### 1.6 Temporally Integrated Monitoring of Ecosystems (TIME) Project

The TIME Project, a long-term monitoring activity, evolves from existing projects within EPA and NAPAP. TIME sites are selected by evaluating data from currently monitored systems and from NSWS results. These sites, which will be established throughout the United States by 1990, are monitored to quantify the rate, direction, and magnitude of changes in surface water chemistry due to increased and decreased levels of acidic deposition. The TIME sites also provide information on surface water chemistry that can be used to validate the conclusions of DDRP, ERP, and the Watershed Processes and Manipulations Project.

### 1.7 Biologically Relevant Chemistry (BRC) Project

The BRC Project provides data that can be used to assess the risk that acidic deposition poses to aquatic biota. Several complementary studies are incorporated as components of BRC. One study is designed to determine the present status of fish populations in a subset of lakes sampled during the eastern component of NLS and quantifies the chemical characteristics of these lakes. Another study, planned in conjunction with ERP, will determine the effects of episodic acidification on fish populations. Initial BRC sampling was conducted from June to September 1987 in the Upper Peninsula of Michigan and northwestern Wisconsin.

### 1.8 Indirect Human Health Effects (IHHE) Project

The IHHE Project targets two areas: (1) the alteration of drinking water supplies in response to acidic inputs and (2) the accumulation of mercury and other potentially toxic metals in the muscle tissues of edible fish. Emphasizing precipitation-dominated surface water systems, drinking water studies include the examination of existing data to determine the potential modification of drinking water quality by acidic deposition. In addition, existing process-oriented and survey data are examined to evaluate the relationship between mercury bioaccumulation in sport fish and surface water chemistry in areas receiving high levels of acidic deposition.

### 1.9 Technical Information Project

The Technical Information Project disseminates information on AERP activities to state and federal agencies, other organizations, and technical audiences. Documentation for several AERP projects is available. All documents can be ordered through the AERP *status*, a periodic update of

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program activities. If you would like to be included on the *status* mailing list, fill out the following form and return it to the address indicated.

Would you like to	be included on the	he mailing list for future editions of the Al	ERP <i>status</i> ?
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If you are on the	mailing list for th	ne AERP <i>status</i> , do you want to remain?	
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### 2.0 Overview of AERP Handbooks

### 2.1 Purpose of Handbooks

Numerous private, state, and federal groups have initiated research projects similar to those developed as components of AERP. Existing AERP field and laboratory manuals and quality assurance plans were not written for an overall methods application or for general use. Developed for specific survey requirements, available operational documents do not provide general guidelines and procedures that can be adapted readily by different research groups. The AERP handbooks are designed to fill this gap. As guidance documents for groups involved in acidic deposition monitoring activities, the handbooks enable researchers to avoid duplication of efforts and to make maximum use of tested methods.

#### 2.1.1 Types of Handbooks

The AERP handbooks focus on surface water chemistry, based on documents written for NSWS, and on soil chemistry, based on DDRP reports. The handbooks contain procedures for field operations, laboratory operations, and quality assurance criteria for water and soil monitoring activities. Surface water chemistry and soil chemistry are discussed in separate three-volume sets.

#### 2.1.2 Structure of Volumes

Because AERP is a dynamic program, each document is contained in a three-ring binder to facilitate inserting additions or modifications. Each document contains an independent Table of Contents with titles, revision numbers, and effective dates of revisions; a complete, updated Table of Contents will accompany dissemination of each revision. The availability of each volume or revision will be announced in the AERP *status*.

### 2.1.3 Interrelationship of Volumes

Each volume of a particular handbook set represents one aspect of an acidic deposition monitoring activity. Collectively, the field, laboratory, and quality assurance handbooks offer a comprehensive guide to surface water chemistry or soil chemistry monitoring.

# 2.2 Content of Field Operations Handbook

This handbook contains procedures for the collection and transportation of surface water samples. These procedures are based on methods used during various stages of NSWS. The field methods described in this handbook have been used for collecting surface water samples of low ionic strength. Detailed procedures for collecting lake water and stream water samples explain the different techniques used when collecting samples by helicopter, by boat, or from shore. The handbook also describes the logistical prerequisites of survey planning and staffing needs.

### 3.0 Survey Planning Considerations

This section discusses planning considerations for field operations. Several aspects of the survey design affect the planning of field operations such as means of access, schedules, base site planning, staffing, protocols, training and safety, and communications. For large and expensive projects, a feasibility survey is recommended to test protocols and logistics. The following discussions emphasize broad-scale, regional surveys because they present particular logistical challenges. However, projects of any size will benefit from consideration of these issues prior to implementation of field operations.

### 3.1 Overview of Field Operations

Several aspects of the survey design have direct bearing on how field operations can be organized and conducted. These include the geographic area covered by a survey, the sampling time frame or "window" in which the survey must be completed, and the measurement and sample requirements of a survey. Projects that cover a large geographic area in a short timeframe (such as lake overturn) require a large number of sampling teams, rapid transportation between sampling points, or both. During the Eastern Lake Survey-Phase I (ELS-I), approximately 2,000 lakes were sampled during fall overturn in the Northeast, Southeast, and Midwest. Helicopters provided rapid transport; their pontoons served as sampling platforms. Twelve two-person sampling teams were employed. Transportation time was reduced by establishing base sites near clusters of lakes to be sampled. Nearly autonomous units, base sites are fully equipped to perform all field operation activities, yet are sufficiently mobile to be relocated after completing activities in a given area. Base site requirements are discussed in Section 3.3.

#### 3.1.1 Measurements

Certain basic measurements (see Table 3-1) in surface waters are common to acidic deposition studies. The number of measurements and the length of time required to perform them, including quality assurance (QA) and quality control (QC) activities, must be considered when determining appropriate sampling schedules and personnel requirements.

### 3.1.2 Sample Holding Time

Sample holding time, defined as the maximum time between sample collection and analysis before detectable changes in the variable of interest can be expected to occur, is a primary consideration in planning field operations. Measures that can extend the holding times of specific variables include eliminating air, refrigerating at 4 °C, freezing in dark storage, or adding chemical preservatives to the sample. Standard reference books for analytical methods, including the Handbook of Methods for Acid Deposition Studies, Laboratory Analyses for Surface Water Chemistry (U.S. EPA, 1987), provide insightful method-specific measures. One way to ensure rapid analysis of variables with short holding times (e.g., pH, dissolved inorganic carbon (DIC), monomeric aluminum) is to locate mobile laboratories near base sites. Another way to achieve rapid sample analysis is by using a centralized processing facility. Samples can be shipped from the field to this central laboratory by overnight courier. Sample processing at the field site is not recommended because there is a risk of contamination for most chemical constituents.

Table 3-1. Variables Measured in Acidic Deposition Studies

In Situ	Field Laboratory	Analytical Laboratory
Connductance Dissolved oxygen Lake Temperature oH Secchi disk transparency	Aluminum, total monomeric and nonexchangeable Dissolved inorganic carbon, closed system Laboratory pH, closed system Specific conductance True color Turbidity	Acid neutralizing capacity Aluminum, extractable Aluminum, total Ammonium, dissolved Base neutralizing capacity Calcium, dissolved Carbon, dissolved inorganic Carbon, dissolved organic Chloride, dissolved Fluoride, total dissolved Iron, dissolved Magnesium, dissolved Nitrate, dissolved pH Potassium, total Potassium, dissolved Silica, dissolved Sodium, dissolved Specific conductance Sulfate, dissolved True Color

#### 3.2 Access

Before any field operation can begin, access considerations must be resolved. The following subsections discuss several access concerns, including gaining permission to sample, determining the proper mode of transportation, determining the sampling platform, and facilitating access during field operations.

#### 3.2.1 Access Permission

Written permission to sample should be obtained from the owner of the surface water to be sampled and from owners of property which must be crossed in order to access the water body. First, all owners must be identified. Various state and local agencies can provide helpful landowner For AERP projects, the Soil Conservation Service proved helpful in identifying Second, landowners should be contacted, either verbally, in writing, or both. Information given to the landowner should be as specific as possible regarding how the water body will be accessed, when, by whom, what will be done, and why. A pamphlet describing the project can be a useful descriptive tool. Landowners may place restrictions on the mode of access. For example, permission was denied for helicopter access to NSWS lakes in wilderness areas, but permission was granted for access by hoofed animal (horses, mules, llamas) or by foot (hiking). Similarly, use of motorized boats was denied for several municipal water system reservoirs. but nonmotorized inflatable craft were permitted. Third, the landowner should sign a written agreement, and the landowner and the sampling team each should receive copies of the agreement. Many landowners may require a waiver of liability or proof of insurance. It is important to remember that not all surface waters are privately owned. Permits may be required to access publicly controlled water bodies. All permits and documentation of access permissions should be completed and filed prior to the initiation of sampling.

#### 3.2.2 Transportation

Unless restricted by access permission agreements, a variety of options are available for transportation. Helicopters equipped with pontoons served as the transport vehicle and sampling platform during ELS-I and WLS. Fixed-wing aircraft were used during WLS to transport samples and supplies between remote base sites and the mobile field laboratories. Other transportation modes include 4-wheel drive vehicles, hoofed animals, foot power, skis, and snowmobiles.

The preferred means of access can sometimes be determined from maps, although a reconnaissance trip is recommended to verify road conditions and to estimate travel times. The reconnaissance trip can also be used to complete site identification and to meet with landowners to finalize access permissions. The season in which field operations take place should also be considered; roads which are passable in summer may be impassable in winter snows or spring rains.

#### 3.2.3 Access Kits

During NSWS, samplers received access kits prior to sampling activities each day: These access kits included lake maps with routes, boat launch areas, and sampling points; copies of the written access permissions; names, addresses, and telephone numbers of landowners and other contacts; pamphlets describing the project; placards to identify the access vehicle; and identification badges for samplers. Landowners were notified one to two days prior to the scheduled sampling date.

#### 3.2.4 Sampling Platforms

Except for streamside sampling of small streams, sampling platforms are generally a necessity. Platforms provide a stable surface from which samplers can operate. Sampling platform options include helicopters with pontoons, fixed-wing aircraft (for large water bodies) with pontoons, solid or inflatable watercraft (with or without motors), permanent floating surfaces, or across-stream rigging. The sampling platform is chosen after considering access permission restrictions, cost restraints, and portability. A reconnaissance trip is also useful to identify launch areas and physical limitations of a particular platform.

#### 3.3 Base Sites

Base sites are a temporary headquarters for localized sampling operations. They are useful for sampling activities that (1) cover a wide geographic area, (2) are distant from the project offices or from the analytical laboratories, (3) have many sites located in clusters, or (4) must be completed during a specific seasonal timeframe. To be effective, base sites must be fully equipped, nearly autonomous units that are also mobile.

Base sites can house personnel involved in field operations, sample processing operations, and some sample analyses. To include sample processing and analysis operations, a mobile field laboratory should be colocated with the sampling teams. Modified trailers were used during NSWS to perform pH, DIC, true color, and turbidity measurements and to prepare chemically stabilized aliquots for subsequent analyses at permanent laboratory facilities. Mobile field laboratories produce the best results if the base site remains in one place for several weeks; they are not as effective if the base site frequently moves. The mobile field laboratories require 2 to 5 days downtime with each move to ensure proper instrument operations. Requirements of mobile

laboratories are discussed in detail in the Handbook of Methods for Acid Deposition Studies, Laboratory Analyses for Surface Water Chemistry (U.S. EPA, 1987).

If helicopters are used as a means of access, the base site must include landing and fueling facilities. Most airports will accommodate helicopters if arrangements are made in advance. During ELS-I and WLS, mobile laboratories were located at or near airport facilities.

Personnel at base sites are usually responsible for the shipment of samples to processing facilities or to analytical laboratories. Most overnight courier services provide a complete listing of their facilities and schedules. If volume is large, it is best to contact these facilities well in advance. In some cases, the courier services may provide special services such as direct sample pickup and delivery at the base site.

The basic facilities of the base site should include:

- 1. An area for instrument calibration.
- 2. Sufficient storage for all instruments, supplies, and related sampling gear.
- 3. A logistics room for office space, conferences, and daily planning and debriefing.
- 4. Ample refrigerator and freezer space for samples, reagents, and frozen gel packs.

Space can be leased or coordinated through local agencies involved in the project. Motels can also serve as base sites; extra rooms or suites can be used for the base site facilities. If motels are used, connecting rooms on the first floor should be requested and maids should be instructed not to use any cleaning solutions in the calibration or storage rooms. Refrigeration and freezer space, if not available through the motel or leased space, can be obtained at meat storage lockers, icehouses, or dairies. Care should be taken to keep sampling gear and calibration solutions separate from food items.

Additionally, it is recommended that accounts be established with local suppliers. While most supplies can be shipped to base sites from a centralized warehouse, some items may be needed more quickly or may be more convenient to purchase on site. Such needs might include clothing and safety gear, small equipment related to sampling, and office supplies. Accounts for vehicle repair and maintenance are also useful.

Personnel facilities and needs include lodging, food, banking services, laundry, and some amenities. Rental houses or condominiums are an alternative to motel lodging and can be more cost effective for sites in operation more than a few weeks. This type of lodging also provides field personnel with an alternative to restaurant dining. Banking services are very important for personnel on travel for more than a couple of weeks.

Emergency services should also be investigated for each base site, including police, fire, hospitals, and search and rescue. Local telephone numbers should be included in the access kits described in Section 3.2.3 and posted next to each telephone within the base site facilities.

Some sampling locations, situated far from an existing base site, may require overnight travel and the establishment of a remote base site. In this case, a sampling team travels to the sampling site, collects the required samples, and ships them from a remote location. The sampling team must carry all needed equipment for calibration, sample collection, and sample shipment (e.g., Table 4-1).

### 3.4 Sampling Schedules

Sampling schedules are developed after a desired sampling window has been chosen. The sampling window is often dictated by climatic conditions, such as lake overturn, stratification, melting ice cover, or spring leafout. While subject to slight variation on a yearly basis, approximate windows can be determined from past climatic data. Windows vary with latitude, elevation, and proximity to coastal areas; therefore, discrete windows should be determined for different ecosystems. Once these windows are identified, a general schedule can be developed to establish a base site relocation scheme.

The following five steps guide the selection of base sites and tentative sampling schedules:

- 1. Identify (mark) each sampling location on a large-scale map (1:250,000 and 1:100,000 scales were used for NSWS) and the type of access to be used (i.e., helicopter, vehicle, foot).
- 2. Identify discrete clusters of sampling points, if possible. If clusters exist, identify the urban areas located within the cluster. Check these urban areas for availability of the facilities needed to establish a base site (Section 3.3).
- 3. Draw concentric circles around potential base site locations. The diameter of the circle should be approximately equal to the maximum distance that a team can sample and return to the base site in one work day. A general guideline for these circles is to allow approximately 100 miles diameter for road travel or approximately 300 miles diameter for helicopter access. Where discrete clusters of sampling points do not exist, it may be necessary to identify all possible base site locations, construct the map circles, and select the best alternative locations based on the maximum number of sampling sites located within the circle.
- 4. Examine each point within the circle and verify that it can be sampled in a single work day. Identify those points that cannot be sampled in a single day due to the lack of roads, long hikes, or other physical limitations. Also examine points lying outside the map circles and verify that they cannot be reached easily from the base site. If possible, construct another base site circle to include these points. If not possible, identify these points as remote sites (overnight travel required).
- 5. After base sites have been selected, tentative routings and schedules should be worked out. During ELS-I, base sites were initially established in the northern area. As each group completed sampling within the base site area, the entire tearn was relocated to a more southern site. A total of 8 base sites was used to complete the survey. In addition, 11 remote base sites were employed during the survey.

Sampling schedules should be flexible to allow for bad weather, equipment malfunctions, difficult access, and other impacts upon schedules. A reasonable amount of downtime should be included in all schedules. Base site relocation schedules should include contingencies for annual variations in climate which may alter the sampling window.

#### 3.5 Field Personnel

#### 3.5.1 Base Site Staff Positions

A base site usually consists of a base coordinator and a number of two-person sampling teams. Additional positions, such as a logistics coordinator, may be necessary depending on the complexity of the project. If helicopters are used, the base site staffing also includes one pilot per helicopter and one mechanic. Mobile laboratory positions are described in the *Handbook of Methods for Acid Deposition Studies, Laboratory Analyses for Surface Water Chemistry* (U.S. EPA, 1987). Each of the standard base site positions is described below.

#### 3.5.1.1 Base Coordinator--

Base coordinators direct field activities in a particular area. The base coordinator's primary responsibility is to ensure a thorough and timely progression of lake sample collection and shipment. Before the field sampling program begins, the base coordinator should select base site locations, compile necessary information on each site, makes advance arrangements, assist in training sampling personnel, schedule the sampling sequence, and assign sites to teams. After the field sampling program begins, the base coordinator:

- 1. Contacts local property owners for access permission, as needed.
- 2. Maintains regular phone contact with sampling crews, local cooperators, and a centralized communications center (Section 3.9).
- 3. Arranges shipping and receiving of samples and supplies.
- 4. Checks data forms and logbooks for legibility and completeness.
- 5. Monitors weather developments.
- 6. Coordinates daily scheduling and makes changes, as needed.
- 7. Initiates search and rescue of the sampling crews, if needed.
- 8. Maintains the project and personnel records.

#### 3.5.1.2 Sampling Teams--

For most surveys, sampling teams composed of two scientists, the team leader and the sampler, are satisfactory. The team leader maintains overall responsibility for the team performance and safety and acts both as sampler and QA representative. The sampler assists the team leader and performs on-site sampling duties. Specific duties of the team leader and sampler are discussed in sections 4.3 and 4.4, 5.3 and 5.4 and, 6.3 and 6.4 for boat sampling, helicopter sampling, and stream sampling, respectively.

### 3.5.2 Specialized Base Site Positions

Other positions in addition to the three previously described may be necessary. Large-scale surveys may require a logistics coordinator to assist the base coordinator with field activities.

Operations involving helicopters require pilots, mechanics, and a ground crew member. A duty officer position is recommended for surveys that involve coordination of multiple government and private organizations or surveys that generate media attention. Extremely complex surveys may require a separate manager or coordinator for each major activity, each of whom report to the overall base coordinator. The WLS is an example of a complex survey which involved helicopter sampling, ground sampling with sample transfer teams, and use of mobile laboratories. Additionally, WLS was a collaborative effort of multiple EPA-research laboratories, regional offices, the U.S. Department of Agriculture Forest Service, and associated contractors. The base site organizational structure of WLS is shown in Figure 3-1. Each of these specialized positions is described below.

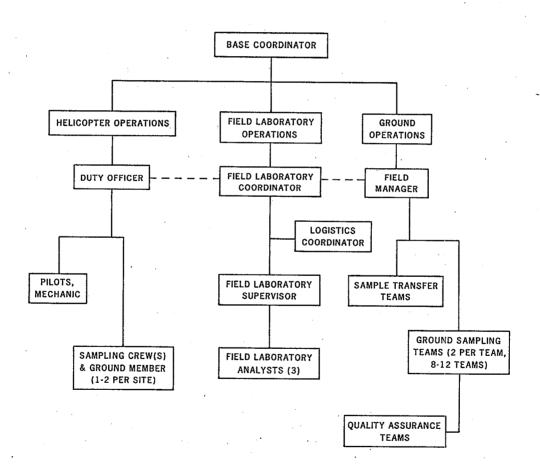


Figure 3-1. Base site organizational structure for the Western Lake Survey.

### 3.5.2.1 Logistics Coordinator--

Especially useful during large-scale surveys, the logistics coordinator assists the base coordinator as needed. In addition, the logistics coordinator provides the following services:

- 1. Coordinates moves between base sites.
- 2. Maintains the supply inventory.
- 3. Verifies that all sampling supplies and access kits are complete.
- 4. Assists in setting up the calibration room.
- 5. Assists sampling personnel when they return from the field; checks the field data forms; assists with post-sampling instrument quality control checks and meter maintenance.
- 6. Checks on road conditions.
- 7. Serves as a substitute sampler.

#### 3.5.2.2 Pilots--

The pilot's primary responsibility is to safely transport field personnel and equipment to and from the preselected lakes, the field site, or other predetermined sites. Pilots report directly to the base coordinator. The pilots are responsible for the following tasks:

- 1. Insuring the safety of the sampling team and other individuals who may be involved with the aircraft.
- 2. Filing a flight plan with Flight Services.
- 3. Filing an internal flight plan with the duty officer and/or base coordinator.
- 4. Arranging refueling at remote refueling stops; these stops are coordinated with the base coordinator.
- 5. Reporting to the duty officer and Flight Services the time of departure at each stop and closing out the flight plan at the end of the day.
- 6. Reporting to the duty officer for briefing on the next day's sampling plan and assisting in route selection for sampling. Each evening, the pilot reviews and plots the next day's sampling route.
- 7. Checking weather prior to take-off.
- 8. Aborting flight plan under unsafe conditions.
- 9. Maintaining an accurate Loran C operation.
- 10. Reading depth sounder to locate sampling site on lake.
- 11. Maintaining position of the helicopter while at the sampling site.

#### 3.5.2.3 Ground Crew Member--

NOTE: These duties may be performed by the base coordinator if the base site supports only one sampling team.

The duties of the ground crew member are dictated by the needs of the helicopter sampling team, duty officer, and base coordinator. Preflight departure activities include:

- 1. Calibrating instruments to be used by the field crew and completing the Hydrolab Calibration Form (Appendix A, Figure A-1).
- 2. Assisting helicopter sampler teams in obtaining, transporting, and loading equipment and supplies for the day's sampling activities.

### Postflight departure activities include:

- 1. Meeting with the duty officer or base coordinator to get lists of lakes to be sampled the following day.
- 2. Organizing all maps for the lakes to be sampled and completing appropriate parts of field data forms, including a sketch of each lake drawn from a U.S. Geological Survey (USGS) 7.5 minute or 15 minute quadrangle map.
- 3. Obtaining required supplies and QC solutions from the field laboratory coordinator, as necessary.
- 4. Completing Lake Coordinates Form (Appendix A, Figure A-2) for next day's sampling sites.

#### Postflight return activities include:

- 1. Rechecking calibration of instruments in use during the day and providing completed calibration forms to the base coordinator.
- 2. Verifying that all equipment and supplies are ready for the next day.
- 3. Having defective equipment repaired or replaced through the duty officer.
- 4. Reporting to the duty officer for debriefing on the day's activities.
- 5. Delivering Lake Coordinates Form to the pilot for next day's sampling site.

### 3.5.2.4 Duty Officer--

The primary purpose of the duty officer position during the ELS-I was to provide a political liason between government agencies and the media. These duties may be performed by the base coordinator. The responsibilities of the duty officer include:

- 1. Coordinating activities of the base site with a centralized communications center.
- 2. Preparing sampling itineraries and flight plans.

- 3. Tracking daily sampling activities via phone check-in (helicopter) or by contact with the base coordinator (ground).
- 4. Tracking progress of sampling via maps and a written log.
- 5. Debriefing sampling teams each day.
- 6. Coordinating maintenance of field equipment and supply requests with sampling personnel and the field laboratory coordinator.
- 7. Assisting the base coordinator with search and rescue efforts.

### 3.5.2.5 Sample Transfer Teams--

Sample transfer teams are additional personnel who accompany the ground sampling team. After collection of samples, the sample transfer personnel transport samples as rapidly as possible to a pre-arranged pick-up point with a helicopter or vehicle. The purpose of sample transfer teams is to reduce the length of time between sample collection and processing at the mobile laboratory. Sample transfer teams were used in the WLS where sampling of lakes in wilderness areas required long hikes with all equipment carried in backpacks.

### 3.6 Sampling Protocols

Standardized protocols are essential to ensure comparability among sample measurements. These protocols should include instrument calibration, quality control checks, measurement procedures, maintenance schedules, troubleshooting guidance, and sample collection procedures. The protocols developed for the AERP projects are described in this handbook. All protocols should be developed, tested, and documented prior to initiation of field operations. Protocols can be developed in conjunction with equipment evaluation experiments. Equipment evaluations are useful to (1) verify manufacturers' specifications of instrument performance, (2) select among various instrument models, and (3) determine instrument limitations.

The written protocols should be assembled into a field sampling manual. Manuals are valuable tools both in training personnel and during sampling operations. This handbook is intended to provide the basis for field sampling manuals. The field sampling manual should also contain copies of all standardized forms, with clear and complete instructions for their completion. Copies of the forms developed for the AERP projects are contained in Appendix A.

### 3.7 Sample Requirements

The types of samples collected are dictated by logistical problems, QA and QC requirements, and survey protocols. During AERP surveys, routine samples, duplicate samples, and blank samples were collected for water chemistry analysis. Specialized samples (e.g., chlorophyll and zooplankton) are discussed in sections 16.0 and 17.0, respectively.

### 3.7.1 Routine Sample

For AERP surveys, a routine sample consisted of one 4-liter (L) Cubitainer and four syringes. Collection procedures are discussed in method-specific sections of this handbook. The syringe

samples were collected for analyses of dissolved inorganic carbon, pH, and two aluminum analyses.

### 3.7.2 Duplicate Sample

A duplicate sample consisted of an additional set of sample containers that were collected immediately following the collection of a routine sample. Generally, one duplicate sample was collected per base site each day.

#### 3.7.3 Blank Sample

Blank samples are deionized water collected from the sampling device using the same method as for the routine sample. Blank samples consisted of a 4-L Cubitainer and two syringes drawn for monomeric aluminum analyses. During NSWS one blank sample was collected per base site each day in accordance with the specifications in the quality assurance plan.

### 3.8 Training and Safety

Recommended qualifications for sampling personnel include a knowledge of basic chemistry or limnology, field sampling experience, a high level of work neatness and precision, and survival and safety skills. As a condition of employment, it is recommended that personnel are certified in cardio-pulmonary resuscitation (CPR) and first aid or that these are subjects included in the training program. A college degree in one of the physical sciences is recommended, but is not absolutely necessary. Outdoor skills and attention to detail are necessary qualifications for field samplers; organizational and management skills are needed by base coordinators. General physical fitness and moderate strength are important qualities in field samplers, particularly if backpacking is required to gain access to sampling sites.

### 3.8.1 Training Program

Training programs for field samplers should include thorough coverage of each procedure and hands-on practice sessions. Table 3-2 lists possible topics to be included in a training program.

Table 3-2. Possible Topics to be Covered in a Training Program for Field Samplers

- 1. Employee orientation; project overview.
- 2. Water sample collection.
- 3. Sample handling, packing, and shipping.
- 4. Instrument operation, calibration, maintenance,
- and packing.Limnology principles and/or stream hydrology and hydrologic measurements.
- 6. Site reconnaissance.

- 7. Data form use.
- 8. Field safety.
- a. First aid and CPR
  - b. Wilderness survival
  - Communications
  - d. Defensive driving; 4-wheel drive training
  - e. Water safety
  - f. Helicopter safety

Theory and rationale for each rule and procedure should be covered in detail; a basic review of limnological principles is recommended. A typical training schedule should include one day of orientation, including explanation of rules and an overview of operations. Another day should be devoted to each method or procedure, including general field procedures such as boat launching.

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Training should include lectures, a demonstration of procedures, question and answer sessions, and hands-on sampling practice. At the conclusion of training, a written test is recommended. The training period also permits assessment of potential sampling teams with complementary skills.

Safety training should include defensive and 4-wheel driving, water and boating safety, advanced first aid, CPR, and survival skills. Specialized safety training is recommended for helicopter sampling. A complete physical examination is recommended; complete medical surveillance may be needed if hazardous materials are used.

For projects conducted in wilderness areas, training in orienteering is important. Map reading skills are vital for field samplers. At least one full day should be devoted to orienteering skills, with subsequent practice and testing.

### 3.8.2 Field Safety

The AERP studies have an excellent safety record, with no on-site injuries. This record is due primarily to the safety precautions included in each project and the emphasis placed on safety throughout training and operations. In addition to the safety training mentioned above, field samplers also should be provided with or required to have protective clothing and sturdy boots. Each team should have a first aid kit with the contents listed in Table 3-3. Table 3-4 lists protective gear that should be provided for each sampling team.

#### Table 3-3. Contents of a First Aid Kit for Field Operations

- 1. Small gauze pads (4)
- Large gauze pad (1)
   Large muslin bandages (2)
- 4. Adhesive bandages (16)
- 5. Eye dressing unit (1)
- 8. Antiseptic unit of providone iodine (1)
- 7. Roll of 2-inch wide elastic bandage (1)
- 8. Roll of adhesive tape (1)

- 9. Ophthalmic irrigation solution
- 10. Aspirin tablets
- 11. Forceps
- 12. Scissors
- Medihaler-Epi (for acute asthma attacks) 13.
- 14. Chlor-Amine tablets (for allergic reaction)
- 15. Instructions for using the above items.

#### Table 3-4. Protective Gear Required for Each Sampling Team

- 1. Tent
- 2. Rain shelter
- Sleeping bags and pads
- Backpacker stove
- Headlamp
- 6. Flashlight
- 7. Backpacker lantem

- 8. Emergency food rations
- 9. Survival saw
- Thermal blanket
- 11. Compass
- Safety line 12.
- Waders
- 14. Waterproof matches.

As a safety precaution during AERP studies, sampling teams filled out an itinerary (Appendix A, Figure A-3) for each sampling day. The itinerary contained proposed routes and schedules, descriptions of samplers' clothing, vehicle identification, and scheduled check-in times. The base and logistics coordinators reviewed each itinerary during the morning briefing and received checkin phone calls from the samplers. Missing a check-in call resulted in initiation of search and rescue activities.

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With the exception of helicopter sampling, field operations involve extensive driving. Potential hazards involving motor vehicles include accidents and mechanical problems. Samplers should be alert when driving and try to avoid these problems. Before driving, samplers should get plenty of rest, avoid medication that causes drowsiness, and file an itinerary form with the base coordinator. Daily travel should be limited to a maximum of 400 miles between two drivers. Table 3-5 lists the maintenance and safety equipment that should be stored in the vehicle.

Table 3-5. Vehicle Maintenance and Safety Equipment

Maintenance Equipment	Safety Equipment
Spare tire Lug wrench Jack and handler Jumper cables Tool kit Tire gauge Spare fuses	Warning triangle or flares First aid kit Fire extinguisher Survival rations Spare change of clothes for each person Shovel Axe or saw

### 3.9 Communications

Communications are vital to any successful field project. In projects involving numerous individuals, it is helpful to have an established chain-of-command so that all participants are aware of their duties and the limitations of each person's authority.

The first level of communications is among the personnel at the base site itself. Contacts should be made with landowners several days prior to sampling to verify previously obtained written permissions and to make arrangements for any obstacles (e.g., locked gates). Daily preand postsampling debriefing sessions are also important to resolve problems, address questions and issues, and discuss any proposed changes in protocols or schedules. When preparing to relocate the base site, it is also helpful to reconfirm all arrangements made for the next site. Schedule changes necessitated by weather or other variables should also be conveyed to all affected parties.

The second level of communications is among base sites. One effective mechanism is a regularly scheduled conference call among base site coordinators. Conference call topics might include problem solving, discussion of protocol changes, supply needs, and discussion of schedule changes.

During NSWS, a centralized communications center provided coordination of all aspects of operations and served as a "clearing house" for all information. As many as six base sites, a processing laboratory, several analytical laboratories, a supplier of audit samples, a sample management office, a quality assurance group, a central warehouse, and multiple levels of management were involved simultaneously in various phases of NSWS.

The base site coordinators were required to contact the communications center twice daily, after sampling teams departed in the morning and after samples were shipped in the evening. The processing laboratory also reported to the communications center twice daily, after field

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samples were logged in and after processed samples were shipped. Analytical laboratories contacted the communications center each day after sample receipt and log-in. In this way, the communications center was able to provide complete sample tracking on a real-time basis so that errors in identification were quickly resolved and samples that were lost or destroyed could be collected again before the base site was relocated. The communications center also handled requests for supplies and relayed the information to the processing laboratory and warehouse. Additionally, the communications center participated in conference calls among base sites and weekly management conference calls. Communications center personnel informed all parties of day-to-day progress and any potential problems. The communications center also maintained documentation on all calls, shipments, and samples. Copies of the forms kept by the communications center are contained in Appendix A, Figures A-4 and A-5.

### 3.10 Pilot Surveys

The purpose of a pilot survey is to test the logistics plan, equipment, and protocols prior to implementation of the full survey. Pilot surveys are recommended for large and expensive programs, state-of-the-art measurements, or programs in which personnel safety issues must be assessed before involving numerous people. In order to be useful, the pilot survey should employ all plans for the full-scale project, including the same organizational positions, access mechanisms, communications network, equipment, and protocols.

### 3.11 References

U.S. EPA (Environmental Protection Agency). 1987. Handbook of Methods for Acid Deposition Studies: Laboratory Analyses for Surface Water Chemistry. EPA 600/4-87/026. U.S. Environmental Protection Agency, Office of Research and Development, Washington, D.C. 342 pp.

## 4.0 Lake Sampling Operations--Boats

#### 4.1 Overview

Boats were used during the Eastern Lake Survey-Phase II (ELS-II) spring, summer, and fall chemistry surveys in 1986. Boats were also used to sample lakes in wilderness areas during the WLS. This section describes lake access planning, sampling activities, and safety considerations related boat operations.

### 4.2 Planning

Planning should begin well in advance of a major survey. Objectives of the project should be clearly stated and a thorough quality assurance plan should be specified. A detailed discussion of planning considerations is contained in Section 3.0.

### 4.2.1 Sampling Teams

During NSWS, each boat team, consisting of two scientists, collected samples from an average of 1.5 lakes per day. During each of the ELS-II seasonal studies, 5 boat teams and 2 helicopter teams sampled approximately 150 lakes in a one-month period (samplers worked Monday through Friday). Helicopter teams are discussed in Section 5.0.

Sampling teams are responsible for loading the vehicle, checking sampling equipment, collecting samples following prescribed protocols, and transferring the samples to the base coordinator upon arrival at the base site. Section 4.3 lists on-site duties of the sampling team and Section 4.4 describes sampling team field operations in detail.

## 4.2.2 Equipment

A complete list of commonly needed equipment for collecting water samples from a boat is provided in Table 4-1. This list should be checked daily during field operations to ensure that all equipment is packed prior to departure to the lake site. Inventories of consumable items should be monitored daily and replenished as needed.

When possible, spare parts and equipment should be maintained at the base site and carried into the field to minimize wasted time caused by malfunctioning equipment. The sampler should notify the team leader of any equipment malfunction immediately.

#### 4.2.3 Lake Access

Written permission for lake access should be obtained prior to initiation of sampling. Copies of access agreements should be maintained at the base site in packets containing maps and data forms; other copies should be carried into the field to aid in mitigating possible access disputes. Base coordinators should inform private landowners of the actual sampling date at least 24 hours

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prior to arrival. The sampling team is responsible for maintaining good public relations in the field. Under no circumstances should a vehicle be driven into an area where motorized vehicles are prohibited. Section 3.2. discusses the procedure for obtaining access.

Table 4-1. Field Sampling Check List for Sampling Water From Boats

#### I. Regular sampling

#### A. Hydrolab Gear:

- 1 Hydrolab Surveyor II sonde unit with
- storage cup or equivalent Calibration cup with cover
- 1 Circulator assembly with cage and weights
- 1 50-meter cable
- 1 Display unit 2 - Batteries
- 1 60-quart hard cooler
- 1 Maintenance kit
- 1 Moisture retardant spray
- 3 Calibration solutions: H<sub>2</sub>SO<sub>4</sub> (0.0001N) 147 μS/cm KCI deionized water

#### B. Water Sample Collection Gear:

- 1 Secchi disk with sounding line weight
- 2 Van Dom samplers with messengers, syringe fitting
- \* Sample kits (Cubitainers, syringes, labels)
- \* Soft coolers
- \* Frozen gel packs
- \* Syringe valves
- \* Syringe protective cases (1 per sample)
- \* Lake data forms
- \* Deionized water for rinsing
- \* Deionized water for blanks (when applicable)
- \* Latex surgical gloves

#### C. Miscellaneous Equipment:

- 1 Clipboard
- 2 Waterproof markers
- 2 Pens
- 2 Pencils
- 1 Field thermometer
- 1 Field notebook

#### C. Miscellaneous Equipment (continued):

- 1 Safety kit
- 1 First aid kit
- 1 Strapping tape
- 1 Knife
- 1 Tool kit
- \* Topographic maps
- \* Calibration forms
- \* Kimwipes (box)
- 1 Flashlight
- 2 Head net
- 2 Insect repellant
- \* Waterproof matches
- \* Drinking water
- \* Sunscreen
- \* Maps
- \* Emergency phone numbers
- \* Sealable plastic bags
- \* Identification

#### II. Remote Sampling

- 1 CO<sub>2</sub> tank 1 CO<sub>2</sub> regulator with Tygon tubing and airstone
- 1 Barometer/altimeter
- 1 Calculator
- 1 NBS-traceable thermometer
- 2 Extra batteries with chargers
- 1 Ring stand
- 1 Ring stand clamp \* Kimwipes (box)
- 1 pH 4.00 buffer (5 gal)
- 1 pH 7.00 buffer (5 gal) 1 KCl (3 Molar)
- \* Deionized water
- \* Lake data forms
- \* Calibration forms
- \* Field communication sheets
- Contact sheets
- \* Shipping coolers

### 4.2.4 Training

All personnel should have a thorough understanding of survey procedures and protocols prior to the implementation of field activities. Training should include lectures on survey objectives, instructions on safety and first aid, and several practice sampling runs. Personnel should discuss any new problems, questions, and concerns that may develop during these training sessions. Training is discussed in Section 3.8.

<sup>\*</sup> Will vary according to sample load.

### 4.3 Field Personnel

Field personnel at each base site include a base coordinator and at least one boat sampling team. The sampling team consists of two samplers. The duties of the base coordinator are discussed in detail in Section 3.5.1.1. The on-site sampling duties of the sampling team are shown in Table 4-2.

### 4.4 Field Operations

The following discussion of methods for field operations is drawn from lake sampling activities during the spring, summer, and fall seasonal periods for ELS-II. Additional specialized seasonal sampling activities may include collecting a sample for nitrate/sulfate analysis, anoxic iron and manganese, chlorophyll  $\alpha$  samples, and zooplankton samples. These activities are described in sections 14.0 through 17.0.

In all of the sampling periods, one sample is taken from the deepest part of the lake. This sample includes four syringes and one 4-L Cubitainer. Duplicate samples and field blanks are also collected.

Complete temperature and conductivity profiles should be taken if the lake is stratified. Secchi disk measurements should be taken before completion of the profile. Standard operating procedures should be separated into predeparture, en route, arrival at sampling site, onshore, and postsampling activities. Each component is described in this section. The sequence of operations during each activity is outlined in Table 4-2.

### 4.4.1 Predeparture Activities

Prior to departure from the base site, Sampler #1 calibrates the Hydrolab and performs a quality control check. Specific procedures for Hydrolab calibration are given in Section 7.1.2. Sampler #2 loads the equipment and supplies. Meters, probes, and other sampling gear should be packed so as to minimize physical shock and vibration during transport. Sample containers (Cubitainers, syringes) are prepackaged into kits for each lake to minimize contamination potential. Additional kits for assigned quality assurance samples and an extra kit for spare supplies should be packed also. Two 4-L Cubitainers of deionized water are required for each field blank to be collected.

The sampling team must set a sampling itinerary prior to departure (see Appendix A, Figure A-3). This itinerary should include departure time, estimated duration of excursion, proposed call-in schedule, route of travel, location of any overnight stops, and the estimated time of arrival at the final destination (base site or other designated sample pick-up point). The base coordinator initiates search and rescue measures if samplers miss designated call-in times.

#### 4.4.2 Arrival Activities

Upon arrival at the designated lake, the sampling crew should verify the proper identification of the lake. This can be accomplished by (1) comparing the lake shape to that shown on a USGS 7.5-minute map, (2) confirming the lake position relative to topographic features shown on the map, or (3) receiving assistance from a local person familiar with the area.

Table 4-2. Duties of Boat Sampling Teams

Duties	Sampler No. 1	Sampler No.2
Predeparture:	-calibrates Hydrolab -inspects batteries	-performs final review of maps and access routes -completes field itineraries -reviews equipment checklist -loads vehicle and boat for travel (tires, keel, pressure, electrical connections, tie down, motor position, etc.)
En Route to Lake:	-drives to site	<ul> <li>-navigates (furthest lake first)</li> <li>-fills out field notebook (mileage, notes)</li> </ul>
Arrival at Lake:	<ul> <li>-verifies correct sampling site</li> <li>-performs field quality control check sample on Hydrolab</li> <li>-navigates to sampling site</li> </ul>	-checks boat safety -loads equipment into boat -pilots boat
At Sampling Site:	-determines anchor drop -records data -records meteorological site informa- tion -determines Secchi disk transparency -equilibrates sonde unit -lowers sonde unit through water column -attaches valves, neck labels, clear- air bubbles -place samples on gel packs	-operates display unit -determines lake strata depths -organizes sample kits -prepares Cubitainers, syringe labels -drops Van Dorn sampler to 1.5 m -fills syringes and Cubitainers -prepares Van Dorn sampler for sample collection (runs blank, if needed) -gives aliquots to Sampler #1
REPEAT SA	AMPLE COLLECTION PROCEDURES FOR D	UPLICATES
Onshore:	-transfers samples to hard cooler -unloads boat	-helps unload boat
	SECURE BOAT AND VEHICLE FOR TRAVE	L
Proceed to Next Lake or Base Site:	-drives .	-navigates -transcribes data and access information
Back at Base Site:	-performs Hydrolab QC check -reviews forms for transcription errors -transfers data forms to base coordinator	-prepares samples for transfer to base coordinator -completes lake data forms

After the lake is positively identified, the site description portion of the Lake Data Form (Appendix A, Figure A-6) should be completed. The method of verification should be documented in the "Comments" section of the data form.

NOTE: If weather conditions are unsafe, sampling should be suspended. Sampling can be done in a light rain, with the sampling personnel protecting the sample from rain contamination. No insect repellant or other contaminant should be on the hands of the sampling crew. Disposable, nonpowdered latex gloves should be worn while sampling.

The sampling crew travels by boat to the designated location (e.g., the deepest part of the lake) marked on the lake sketch. If conditions permit (lack of wind, shallow water), the boat should not be anchored. If the position cannot be maintained at the sampling location, the boat should be anchored at a location well upwind of the sampling location. The boat should then be permitted to drift over the sampling site, and the anchor line should be secured.

### 4.4.3 Activities At Sampling Site

After the boat is secured, the site depth should be measured. The site depth is determined by lowering a Secchi disk on a calibrated line into the water. The site depth should be printed on the data form. The second measurement to be taken should be the Secchi disk transparency determination (Section 11.0). This measurement should be made in the shade of the boat; the sampler must not wear sunglasses. The Secchi disk is slowly lowered on a marked line until it disappears from view; this depth should be recorded on the data form. Then the disk is raised slowly until it just reappears; this depth is recorded on the data form also. The Secchi disk transparency, calculated later, is the average of the two recorded depths.

Stratification status is determined next. The Hydrolab should be allowed to equilibrate in the lake for approximately five minutes. Then temperature, pH, dissolved oxygen (DO), and conductivity should be measured at 1.5 meters (m) below the lake surface and at 1.5 m above the lake bottom. If the temperature between these two depths is greater than 4 °C, the Hydrolab should be raised to 0.6 of the site depth and temperature, pH, DO, and conductivity should be measured again. If there is still a greater than 4 °C difference between the 1.5 m depth and the 60 percent lake depth, the lake is considered to be stratified and a vertical profile of temperature and conductivity should be made. Specific measurement depth intervals and profiling procedures are discussed in Section 7.0.

After the stratification profile is completed, the blank sample, routine sample, and duplicate sample should be collected with a Van Dorn sampler as described in Section 12.0. Four syringes (one for DIC, one for pH, and two for monomeric aluminum analyses) and one Cubitainer should be filled from this sample. Blank samples and duplicate samples also should be collected through the Van Dorn sampler. A duplicate sample is a second sample collected immediately after the routine sample. When collecting a blank sample, the Van Dorn sampler should be rinsed with three separate 200-milliliter (mL) volumes of deionized water, then filled with deionized water and sealed. Two syringes (for monomeric aluminum analyses) and one Cubitainer should be collected from this sample. Specific operating procedures for the Van Dorn sampler and for sample collection procedures should be covered thoroughly during training.

When finished with measurements and collection procedures, the boat team must:

- 1. Coil all lines and cables neatly, avoiding kinks in the cable.
- Rinse the Hydrolab sonde with lake water and replace the storage cup filled with tap or lake water for transport.
- 3. Empty the Van Dorn sampler of excess water and secure it for travel.
- 4. Replace and secure all gear.
- 5. Leave Hydrolab cables connected unless absolutely necessary to disconnect. (Connections should be relubricated weekly to ensure that leakage does not occur.)

Before the team leaves the lake site, Sampler #1 should verify that all forms and labels have been properly completed and that all required samples and parameters have been taken or measured.

#### 4.4.4 Onshore Activities

Samples should be stored at 4 °C to minimize biological or chemical changes in the sample until they are delivered to the laboratory. After the sampling procedures have been completed, the boat is returned to shore. Onshore, the syringe valves are checked to be sure they are closed and that no airspace exists in the syringes. The syringes should be placed into a holding container (a food storage container of suitable size was used on all NSWS surveys) to minimize disturbance and possible leakage. A 30-quart ice chest can be lined with frozen gel packs enclosed in sealable plastic bags. The Cubitainers should be placed in the center of the ice chest, and the syringe container should be placed on top of the Cubitainers. If possible, all samples collected from an individual lake should be packed in the same ice chest.

All information in the field logbook should be transcribed to the proper forms by Sampler #2. Sampler #1 should check all transcribed information for potential errors. The completed forms should be enclosed in a sealable plastic bag for transport.

### 4.4.5 Postsampling Activities

At the base site, the ice chests and forms should be transferred to the base coordinator who prepares the samples for shipment. Sampler #1 postcalibrates the Hydrolab (Section 7.0), and Sampler #2 completes the necessary forms.

### 4.5 Boat Safety

There are several safety precautions related to the use of boats.

NOTE: See Section 3.8.2 for additional safety discussion.

### 4.5.1 Boat Trailer Hauling

When preparing to haul a boat from one location to another, the following instructions should be followed:

- 1. Lower trailer tongue onto ball, making sure the tongue seats properly on the ball. Then fasten the safety latch or bolt. If this is difficult, the ball and tongue may not be joined properly.
- 2. Plug in the connector for the trailer brake lights and turn signals. Have an observer stand behind the vehicle while the driver applies the brakes and turn signals to assure all lights are in working order. When launching the boat from a boat ramp, be sure to unplug this connector before the trailer goes in the water.
- 3. Connect safety chains; allow for proper slack.
- 4. Double check all connections and lights. Inspect these connections frequently during transport.

### 4.5.2 Towing Precautions

Safety precautions related to towing the trailer and boat include the following:

- 1. Do not load too much weight or load weight unevenly into the boat. A capacity label on the trailer lists the gross trailer weight. No more than 10 to 15 percent of the total weight of the trailer should be distributed as the tongue weight (most heavy gear carried in the trailer should be placed over or near the trailer axle).
- 2. Be careful when backing up. Use an observer for guidance if the line of sight is obscured.
- 3. Reduce speed accordingly when approaching dips, bumps, or generally rough road.
- 4. Be particularly careful when driving in bad weather such as wind, snow, rain, or ice. If the trailer starts to "fishtail," let up on the gas but *do not apply the brakes*.
- 5. When being passed by large vehicles, maintain speed or accelerate slightly to keep trailer sway to a minimum.
- 6. Check trailer tires daily. Underinflation is a common problem. Occasionally check the warmth of the trailer hubs when on long drives. If they are hot to the touch, the grease in the wheel bearings may be low.

### 4.5.3 Boating Precautions

When actually making use of a boat on a body of water, observe the following precautions:

- 1. Distribute the load evenly and maintain a low center of gravity within the boat.
- 2. There should be a personal flotation device in the boat for each person. Wear this at all times when in the boat.
- 3. Make sure a fire extinguisher is in each boat.
- 4. If lightning occurs, return to vehicle or take shelter. Also, head for shore if heavy winds or rough water are interfering with safe boating.
- 5. If the boat becomes swamped, remain with the boat. If the water is cold, try to get as much of your body out of the water (by propping yourself up on the boat) as possible. Water conducts heat away from the body 25 times faster than air.

NOTE: Boating safety should be an integral part of training.

## 5.0 Lake Sampling Operations--Helicopters

#### 5.1 Overview

For sampling lakes in remote areas of the United States, AERP surveys have relied on helicopters to gain access and to serve as sampling platforms. The ability to sample a large number of lakes makes the use of helicopters advantageous when the sampling window is of relatively short duration. This section describes lake access planning, sampling logistics, and safety considerations necessary for helicopter operations. The procedures described are similar to those used during ELS and WLS.

### 5.2 Planning

Planning should begin well in advance of a major survey. Objectives of the project should be clearly stated and a thorough QA plan specified. A detailed discussion of planning considerations is contained in Section 3.0.

### 5.2.1 Sampling Teams

During ELS and WLS, helicopter teams, which consisted of the pilot and two scientists (an observer and a sampler) collected samples from an average of six lakes per day. During ELS-I, 7 helicopter teams sampled 1,612 lakes in a 2-month period. During WLS, 5 helicopter teams, along with several boat teams, collected samples from 757 lakes in mountainous terrain in a 2-month period.

Sampling teams supported by a ground crew person, are responsible for loading the helicopter, checking sampling equipment, collecting samples following prescribed protocols, and transfering samples to the ground crew member when the teams arrive at the airport. Their activities are controlled by a duty officer or base coordinator, and, when in the helicopter, by the pilot. Duties of the sampling team are separated into preflight, in-flight, on-lake, and postflight activities. Section 5.4 describes these duties. Duties are performed in accordance with approved methodology and QA and QC plans.

### 5.2.2 Equipment

Helicopter sampling teams should have the same basic equipment requirements as boat sampling teams. Three additional items can be used to verify lake location, document the lake sampled, and determine site depth. A LORAN C latitude/longitude locater, a 35mm camera, and a depth finder are recommended, but are not essential for most sampling operations. Additional safety equipment is required also. Field checklists (Table 5-1) should be used daily to ensure that all equipment is packed prior to departure to the lake site.

Table 5-1. Field Sampling Check List for Helicopter Sampling

#### I. Regular Sampling

#### A. Hydrolab Gear:

- 1 Hydrolab Surveyor II sonde unit with storage cup or equivalent
- Calibration cup with cover
- 1 Circulator assembly with cage and weights
- 1 50-meter cable
- 1 Display unit
- 2 Batteries
- 1 60-quart hard cooler
- 1 Maintenance kit
- 1 Moisture retardant spray
- 3 Calibration solutions: H<sub>2</sub>SO<sub>4</sub> (0.0001N) 147 μS/cm KCl deionized water

#### B. Water Sample Collection Gear:

- 1 Secchi disk with sounding line weight
- 2 Van Dorn samplers with messengers, syringe
- \* Sample kits (Cubitainers, syringes, labels)
- \* Soft coolers
- \* Frozen gel packs
- \* Syringe valves
- \* Syringe protective cases (one per sample)
  \* Lake data forms
- \* Deionized water for rinsing
- \* Deionized water for blanks (when applicable)
- \* Latex surgical gloves

#### C. Miscellaneous Equipment:

- 1 Clipboard
- 2 Waterproof markers
- 2 Pens

#### C. Miscellaneous Equipment (continued)

- 2 Pencils
- 1 Field thermometer
- Field notebook
- 1 Safetv kit
- 1 First aid kit
- 1 Duct tape
- 1 Strapping tape
- 1 Knife
- 1 Tool kit
- Topographic maps
- \* Calibration forms
- \* Kimwipes (box)
- 1 Flashlight
- 2 Head net
- 2 Insect repellant
- \* Waterproof matches
- \* Drinking water
- \* Sunscreen
- \* Maps
- \* Emergency phone numbers
- \* Sealable plastic bags
- \* Identification

#### II. Additional Helicopter Equipment

- 2 Helmets with communications connectors
- 2 Life vests
- 2 Flight suits (Nomex)
- 2 Gloves (Nomex or neoprene mittens and cotton work gloves (optional))
- 1 Safety harness
- 1 Tether line (10 feet)
- 2 Carabiners
- 2 Sleeping bags
- 2 Change of clothing appropriate for weather and terrain

#### 5.2.3 Lake Access

Written permission for lake access should be obtained prior to initiation of sampling. Procedures for obtaining lake access are discussed in Section 3.2.

#### 5.2.4 Training

In addition to thorough training in survey procedures and protocols (see Section 3.8), all personnel flying in helicopters must have received proper flight safety training in classroom and on on-site programs. Helicopter training includes a study of flight safety and practice with individual sampling devices. One or two practice runs are recommended to ensure proper sampling procedures are followed. After practice sessions, all personnel involved with the program should

<sup>\*</sup> Will vary according to sample load.

discuss any new problems, questions, and concerns that may develop during training sessions. Helicopter safety is discussed briefly in Section 5.5 and in detail in Appendix B.

### 5.3 Field Personnel

Field personnel at each base site should include a base coordinator, a ground crew member, and at least one helicopter sampling team. If there is only one helicopter sampling team, the base coordinator may assume the additional responsibilities of the ground crew member. The sampling team consists of the pilot and two scientists, the observer and the sampler. The duties of field personnel, including those in specialized base site positions, are discussed in Section 3.5. This section briefly describes the on-site sampling duties of the sampling team.

Sampling team duties should be divided between the observer and the sampler. The observer sits in the front of the aircraft and is responsible for final identification of the lake and recording of field data on the lake data form (Appendix A, Figure A-6). The sampler, stationed in the rear of the helicopter, collects the samples and makes the necessary field measurements following established protocols. Both crew members should assist the pilot in locating potentially hazardous conditions (e.g., other aircraft, power lines, boats) throughout the flight. Personnel may rotate between sampling and ground crew duties to reduce boredom and fatigue.

The sampling team checks and loads gear in the morning and transfers samples to the ground crew member in the afternoon. Figure 5-1 illustrates helicopter sampling team activities. Section 5.4 describes field operations of the helicopter sampling team.

### 5.4 Field Operations

Standard operating procedures should be separated into preflight, in-flight, sampling, and postflight activities. Each component is described in this section. The sequence of operations during each activity is depicted in Figure 5-1.

### 5.4.1 Preflight Activities

In preparation for a day of sampling, each helicopter sampling team:

- 1. Receives calibrated equipment and supplies from the ground crew member and verifies completeness against the equipment check list (Table 5-1).
- 2. Loads equipment into the helicopter under supervision of the pilot to assure proper weight distribution. The observer is responsible for determining that all equipment and supplies are on board and in good repair.
- 3. Reports accurate weight of field sampling personnel and equipment to pilot.
- Reports any changes in load weight to the pilot.

#### HELICOPTER SAMPLING TEAM ACTIVITIES

#### PREDEPARTURE ACTIVITIES AT BASE SITE

- 1. Calibration of equipment by Ground Crew Member
- 2. Check list of equipment and supplies
- 3. Load aircraft
- 4. Check list of lakes to be sampled, and confirm location on maps
- 5. File flight plan with Base Coordinator

#### TRAVEL TO LAKE

- 1. Aid in navigation and aircraft observation
- 2. Verify lake identification
- 3. Record site description watershed disturbances
- Photograph lap card and two aerial photographs note azimuths
- 5. Locate suspected deepest portion of lake

#### SAMPLING SITE ACTIVITIES

- Locate acceptable site with depth sounder and record lake depth
- 2. Determine Secchi disk transparency
- 3. Set buoy for site positioning
- 4. Profile conductivity, pH and temperature with Hydrolab
- 5. If necessary, prepare blank sample
- 6. Collect sample from 1.5 m with Van Dorn
- 7. Obtain DIC and pH syringe samples
- 8. Transfer remaining sample to 4-liter container
- 9. If necessary, prepare a duplicate sample
- 10. Verify that forms and labels are correct and complete
- 11. Store samples and equipment for flight
- 12. Depart from lake

LAST LAKE OF EXCURSION?

YES

RETURN TO BASE SITE

- 1. Unload samples and equipment
- 2. Check calibration on Hydrolab-record on lake data form
- 3. File lake data forms with Ground Crew Member
- 4. Attend debriefing with Base Coordinator or Duty Officer
- 5. Plan and prepare for next day's sampling

### 5.4.2 In-flight Activities

Upon departure from the field base site, the observer assists in navigation, using maps and aerial photographs. Upon arrival at the lake, the observer confirms the lake's identification and after sampling directs the pilot to the next lake to be sampled.

If the observer verifies the lake in question is the proper lake to be sampled, he conveys this message to the pilot and enters site description information on the lake data form. The pilot has the ultimate decision-making power and responsibility for determining suitable conditions for landing. His judgment is based on such considerations as weather conditions, amount of fuel remaining, and physical hazards.

Upon approach to the confirmed lake, three photographs should be taken and the frames are noted on the lake data form. The first photograph should be the "lap card" to record information about the lake on film. The principal reason for photographing the lake is to confirm that the lake sampled is the correct one. Therefore, the lake morphology is of prime interest. As the pilot circles the lake, two photographs should be taken to document the shape of the lake.

#### 5.4.3 On-lake Activities

After the lake is identified and photographed, there are a number of activities required to complete the sampling tasks. Instructions for the sampling crew would include the following:

- Locate the deepest (approximate) portion of the lake, record the depth, and mark it on the lake sketch. Do not spend more than 5 to 10 minutes on this task. Use general topography and lake morphology as a guide to direct the pilot to expected deep areas. The pilot, through use of a depth sounder, will taxi until a deep area acceptable to the team observer is located. A preferred site is one with a relatively smooth contour so that sondes and sounding lines are not snagged.
  - If the lake is multilobed or dendritic and the location of the best sampling spot is uncertain, the observer selects the best location, keeping in mind that a representative sample is the ultimate objective. In the case of a multilobed or dendritic lake, the largest, deepest, and most downstream section should be selected. Influences from major inflows or localized watershed disturbances (e.g., erosion, clear cutting) should be avoided.
- 2. Set a buoy for site positioning by the pilot. Lower weighted line to the bottom and tie off a line at the buoy. The helicopter may position itself approximately 10 to 20 meters away for the best pilot vantage point; alternately, the helicopter may be positioned with the buoy between the pontoons or with the buoy immediately in front of the helicopter. The pilot maintains the sampling position by visual contact with the buoy and by constant readout from the electronic depth sounder. The observer then converts site depth to meters. This aids the sampler in avoiding the bottom with the Hydrolab sonde when monitoring the bottom minus 1.5 m. The Hydrolab cable should be marked in 1-m increments.
- 3. Determine Secchi disk transparency as described in Section 11.0.
- 4. Take Hydrolab measurements to establish presence or absence of lake stratification as well as to charactorize the lake limnologically. Refer to Section 7.0 for operation of the Hydrolab unit.

- 5. Collect field blank sample, routine sample, and duplicate sample with the Van Dorn sampler as described in Section 12.0.
  - a. If the lake is less than 3 m deep, try to obtain a clean (debris-free) sample from 1.5-44 m with the Van Dorn sampler.
  - b. If a clean sample cannot be obtained from 1.5 m, rinse the sampler and lower it to 1.0 m.

NOTE: Approximately 0.9 m of depth is required to allow for clearance of the stoppers when triggered to avoid entrapment of air or bottom debris.

- 6. Prepare to leave the lake when finished with measurements and collection procedures. The helicopter team must:
  - a. Coil all lines and cables neatly, avoiding kinks in the cable.
  - b. Replace the Hydrolab sonde storage cup filled with tap or lake water for transport.
  - c. Empty the Van Dorn sampler of excess water, close all valves, and secure it for travel.
  - d. Replace and secure all gear.
  - e. Do not disconnect Hydrolab cables unless absolutely necessary. Periodic (once a week) relubrication of connections will assure that leakage does not occur.
- 7. Verify (the observer) that all forms and labels are properly completed and that all required samples and parameters have been taken or measured before the team leaves the lake site.

## 5.4.4 Postflight Activities

After leaving the lake, instructions for the sampling team include the following activities:

- 1. Transfer samples and forms (verified and signed by the observer) to the ground crew member upon arrival at the airfield.
- 2. Report any problems with equipment or samples to the ground crew member who will notify the base coordinator and the duty officer, as appropriate. Document all equipment problems and corrective actions.
- 3. Brief the duty officer and base coordinator on the day's activities and report any problems or suggestions.
- 4. Review the next day's sampling plan.

### 5.4.5 Flight Operations

Helicopter pilots must file required Federal Aviation Administration (FAA) flight plans and safety plans. The pilots then proceed to their aircraft and prepare for takeoff. A typical flight day, weather permitting, may be from 7:00 a.m. to 4:00 p.m. At each refueling stop, the pilot or crew member reports to FAA Flight Services and to a field communications center. In the event that an

aircraft is overdue, search and rescue is initiated automatically by FAA Flight Services 30 minutes after the designated flight plan closure time. The duty officer then maintains contact with the local FAA Flight Services. The FAA Flight Services number should be on the forms provided daily to the pilots.

# 5.5 Helicopter Safety--General Safety Precautions

- 1. Helicopter operations must comply with the applicable general safety rules for aerial operations and practices prescribed by federal and state standards, and, if Office of Aircraft Services (OAS) helicopters are used, by OAS standards.
- 2. Only authorized personnel should be allowed to board the helicopters. Authorization is determined by base coordinators and, ultimately, by the pilot.
- 3. If OAS aircraft are used, that agency provides safety training. This training is mandatory for ground crew members, sampling teams, and alternate samplers. Training should include:
  - a. An audio-visual presentation on helicopter safety and ditching survival.
  - b. A lecture by a trained individual on helicopter safety and personal protective equipment and a general orientation on helicopter capabilities and limitations.
- 4. The pilot is responsible for the safety of the helicopter and passengers at all times.

NOTE: Appendix B provides detailed safety instructions for helicopter operations.

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## 6.0 Stream Sampling Operations

### 6.1 Overview

The sampling program described in this section is patterned after the one used during Phase I of the NSS. In that survey, more than 450 stream reaches were sampled at both an upstream and a downstream location during the spring baseflow period. The locations at which streams were sampled were chosen on a statistical basis, without regard to accessibility. Consequently, many streams were difficult to locate and sample. Because helicopters were not suitable for accessing low order streams, samplers drove 4-wheel drive vehicles as close to stream sites as possible and hiked in with supplies and backpacked out with samples.

### 6.2 Planning

Planning should begin well in advance of a major survey. Objectives of the project should be clearly stated and a thorough QA plan specified. A detailed discussion of planning considerations is contained in Section 3.0.

### 6.2.1 Sampling Teams

Sampling teams of two people increase safety and assure that all necessary equipment can be transported.

Experience gained during NSS-I indicates that one two-person team can collect samples from an average of seven streams (at both an upstream and downstream location) during the course of a 5-day work week. For these surveys, it was assumed that samplers can reach streams within a 50-mile radius of the base site.

Sampling teams should be responsible for loading the vehicle, checking sampling equipment, collecting samples following prescribed protocols, and transferring the samples to the base coordinator upon arrival at the base site. Duties of the sampling team are outlined in Section 6.3. Section 6.4 describes field operations in detail for collecting samples from streams.

### 6.2.2 Equipment

Table 6-1 lists recommended equipment and supplies for obtaining water samples from streams. Sampling teams should check this list each day prior to sampling activities.

#### 6.2.3 Stream Access

Written permission to sample should be obtained from the owner of the stream site to be sampled and from owners of property which must be crossed in order to access the water body (see Section 3.2). Base coordinators should inform private landowners of the actual sampling date at least 24 hours before arrival. Under no circumstances should a vehicle be driven into an

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Table 6-1. Field Sampling Checklist for Stream Sampling

#### I. Regular Sampling

#### A. pH Measurement

- 1 pH meter/case
- 2 Electrodes
- 1 ATC probe
- 3 250-mL beakers

- 2 pH 7.00 buffer (250 mL bottle) 2 pH 4.00 buffer (250 mL bottle) 2 H<sub>2</sub>SO<sub>4</sub> solution 0.0001N (250 mL bottle)
- 1 Electrode filling solution
- 1 Instruction manual
- 1 Stopwatch
- 2 Extra batteries

#### B. Conductivity

- 1 Meter and case
- 2 Extra batteries
- 1 Probe and storage bottle
- 2 74 µS QCCS (250 mL bottle)
- 1 Instruction manual

#### C. Dissolved Oxygen

- 1 Meter and case
- 2 Extra batteries 1 - Probe and bottle
- 1 Calibration chamber
- 1 Membrane kit/filling solution
- 1 Instruction manual

#### D. Team Gear

- \* Stream information packets
- \* Maps
- 1 Field logbook
- \* Forms
- 1 Pump
- 2 Batteries and cable
- \* Sample kits (Cubitainers, syringes, labels)
- \* Syringe protective cases
- \* Sampling boom
- \* Extra sample labels
- \* Sealable plastic bags
- 2 Pens, pencils, marker
- 1 Surveyor's tape
- \* Deionized water for blanks (when applicable)

#### D. Team Gear (continued)

- \* Deionized water for rinsing
- 1 Calculator
- Portable cooler
- \* Large cooler
- \* Frozen gel packs
- \* Latex surgical gloves
- 1 Kimwipes (box) 1 Camera, film, batteries
- 1 Clipboard
- 1 Compass
- 1 Knife
- 1 Correction factor tables
- 1 Staff gauge
- 1 Steel rod
- 1 Mallet
- 1 Flow meter, batteries
- 2 Waders
- 1 Tarp
- \* Ductape
- \* Strapping tape
- \* Emergency phone numbers
- \* Sunscreen
- \* Drinking water
- \* Insect repellant

#### E. Personal Gear (per person)

- 1 Rain gear (coat and pants)
- 1 Snake guards or gaiters
- 1 Flashlight
- 1 Nylon line
- 1 Space blanket
- 1 First aid kit
- 1 Emergency rations
- \* Matches (waterproofed)

#### II. Additional Vehicle Equipment

- 1 Spare tire, lug wrench, jack
- 1 Jumper cables
- 1 Tool kit
- 2 Spare fuses
- 2 Flares
- 1 Fire extinguisher
- 1 Axe/machete 1 - Shovel
- 1 First aid kit
- 1 Spare keys/magnetic case

area where motor vehicles are prohibited. The sampling team should be responsible for maintaining good public relations in the field.

Prior to initiation of sampling operations, a reconnaissance dossier should be compiled for each stream site. The dossier should include (1) topographic maps, highway maps, county maps, and other useful maps; (2) a description of access routes (roads, waterways, and foot trails);

<sup>\*</sup> Will vary according to sample load.

(3) access permits (if required) and names of local contact persons who are familiar with the site or who must be contacted in order to gain access.

In addition to the general training described in Section 3.8, stream sampler training also should include river safety lectures, map and compass instruction, and streamside practice sessions. Sample handling, sample shipping, and completion of forms are emphasized. Experienced scientists should monitor teams during training to ensure consistent techniques among teams.

### 6.3 Field Personnel

Field personnel at each base site include a base coordinator, a logistics coordinator, and a number of sampling teams. The duties of coordinators are described in Section 3.5. Sampling teams consist of two scientists each. Their daily responsibilities include:

- 1. Providing the base coordinator with a daily itinerary of sites to be sampled, routes to be followed, and descriptions of team members and their clothing.
- 2. Conducting the initial and final calibration and QC checks of pH (Section 8.0), conductance (Section 9.0), dissolved oxygen (Section 10.0), and flow meters (Section 6.4.6).
- 3. Traveling from the base site to the identified sampling sites.
- 4. Describing the stream site in question and transcribing the description to the appropriate form (Appendix A, Figure A-7).
- 5. Taking three pictures at each site (a "Lap Card" which lists sampling date and time, stream name and identification (ID), frame number of the lap card, and sampling team ID; a picture looking upstream from the sampling location; and a picture looking downstream from the sampling location).
- 6. Marking the exact sampling location on a USGS 7.5 minute map.
- 7. Operating the peristaltic pump and the sampling boom to collect the Cubitainer and syringe samples, as described in Section 13.0.
- 8. Measuring the in situ temperature, as described in Section 9.0.
- 9. Measuring the in situ conductivity, as described in Section 9.0.
- 10. Measuring the in situ dissolved oxygen, as described in Section 10.0.
- 11. Measuring the pH at streamside, as described in Section 8.0.
- 12. Recording all the sampling data on a stream data form (Appendix A, Figure A-8).
- 13. Taking hydrologic measurements and recording them on a Hydrologic Data form (Appendix A, Figure A-9).
- 14. Checking all data forms for completeness, accuracy, and legibility.
- 15. Preparing samples for shipment or transfer to the processing laboratory.

- 16. Maintaining communications with the base coordinator.
- 17. Performing final quality control checks for the pH meter (Section 8.7.2) and conductivity meter (Section 9.7.2).
- 18. Transferring custody of all samples to the base coordinator.
- 19. Attending a debriefing meeting daily to review activities and problems and to prepare for the following sampling day.

### 6.4 Field Operations

The following discussion describes stream sampling activities.

### 6.4.1 Preparation for Sampling

- Prepackage sample containers and pump tubing in sealed plastic bags to prevent contamination.
- 2. Use indelible pens to mark all Cubitainers with identification information such as stream ID, sample date, sample time, sampling team ID, sampling program, and sample type. In addition, attach a label displaying the same information on the neck of the Cubitainer.
- 3. Do *not* expand Cubitainers before filling them; the weight of the water sample will cause them to expand. Blowing into the Cubitainers to expand them can cause contamination.
- 4. Rinse all sample containers three times with sample water before filling.
- 5. Keep tubing clean before use. If contamination of the tubing is suspected, replace the tubing. If no replacement tubing is available, pump water through the tubing for at least two minutes while the discharge end is immersed in the stream. Note any potential contamination.
- 6. Always have at least two charged batteries available for the peristaltic pump. Rotate the use of these batteries.
- 7. If the peristaltic pump fails to operate, check the battery cable connections, check the battery leads, press the reset button, and replace the battery if necessary.

### 6.4.2 Field Blank Sample Collection

- 1. Place the peristaltic pump on as level a surface as possible.
- 2. Affix the completed labels to one Cubitainer and to two syringes before filling, and mark the labels with the word "Blank."
- 3. Attach a short tubing section to the peristaltic pump, being careful to keep the ends from touching the ground or other contaminating surfaces.
- 4. Rinse the last 6 inches of tubing with deionized water and then immerse in a 4-L Cubitainer of deionized blank water.

- 5. Purge tubing (by using approximately 1/2 Cubitainer of deionized water) to ensure clean-liness. Avoid allowing air bubbles to enter the tubing.
- 6. Turn the pump off. Immerse the intake tubing into the second Cubitainer of deionized water.
- 7. Place a labeled, clean, 4-L Cubitainer under the collection tubing. Do not expand the Cubitainer.
- 8. Turn the pump on and collect 100-200 mL of water in the Cubitainer. Cap and rotate the Cubitainer so that the water contacts all surfaces. Discard the water.
- 9. Repeat the above rinsing procedure two more times.
- 10. Allowing the weight of the water to expand the Cubitainer, collect at least 3 L of deionized water in the Cubitainer. Eliminate all air space and cap the Cubitainer tightly.
- 11. Collect two blank syringe samples by using the method described in Section 6.4.5. These samples are for methyl isobutyl ketone (MIBK)-extractable aluminum and pyrocatechol violet (PCV) aluminum fractions. Rinse the two syringes three times and fill them with deionized water from the pump tubing. Blanks are not collected for DIC or pH syringe samples.

### 6.4.3 Routine Sample Collection

- 1. Place the peristaltic pump on as level a surface as possible.
- 2. Affix a completed label to all the sampling containers before filling them.
- 3. Attach new tubing (10-foot section) to the pump. Leave approximately 20 cm of tubing free. Attach the intake end of the tubing to the sampling wand, leaving 5 cm free.
- 4. Place the intake tubing, with the opening pointing upstream, into a flowing portion of the stream. Immerse the intake tubing to middepth in the flow. Avoid letting the tubing end contact the stream bottom or aquatic vegetation.
- 5. Turn the pump on. Purge the tubing for two minutes. Insert the discharge tubing into the neck of a prelabeled, clean 4-L Cubitainer.
- 6. Collect 100-200 mL of water in the Cubitainer. Cap and rotate it so that the water contacts all the surfaces. Discard the water.
- 7. Repeat the above rinsing procedure two more times.
- 8. Insert the discharge tube approximately 2 to 5 cm into the cubitainer. Turn the pump on and fill the Cubitainer with stream water (do not overfill).
- 9. Eliminate the air space from the Cubitainer; cap it tightly. Place it in the cooler with the frozen-gel packs.

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10. Collect four 60-mL syringe samples as described in Section 6.4.5 and place two syringe samples in each of two sealable plastic bags. Place the bagged syringes in a protective syringe case inside the cooler.

### 6.4.4 Duplicate Sample Collection

NOTE: Do not change the tubing between routine and duplicate sample collection.

- 1. After collecting the routine sample (Section 6.4.3), repeat the procedure with a second 4-L Cubitainer and four additional syringes.
- 2. Label each container "Duplicate."

### 6.4.5 Syringe Sample Collection

NOTE: Four syringe samples are collected for each routine or duplicate sample. Only two syringes are collected for a blank sample. Each routine or duplicate sample syringe is used for one of four analyses:

- 1. pH (not taken for blank)
- 2. Dissolved inorganic carbon (not taken for blank)
- 3. MIBK-extractable aluminum
- 4. PCV aluminum fractions

Since both pH and DIC determinations may be affected by contamination from atmospheric carbon dioxide, it is essential that no outside air contact the samples collected for these determinations. The syringes used for aluminum analyses can be contaminated easily by dust, hands, or any metal objects.

- 1. Prelabel the syringes. The label should be attached so that the milliliter graduations are visible and the label can be read with the syringe tip pointed up and away from the reader.
- 2. Turn the pump on.
- 3. Insert the tip of the 60-mL syringe into the end of the tubing.
- 4. Let the force of the pumped water cause the syringe to fill. Rinse the syringe and discard the water by depressing the syringe plunger.
- 5. Repeat the above rinsing procedure two times.
- 6. Insert the syringe into the tubing again. Collect 60 mL of fresh sample.
- 7. Affix the syringe valve. Close the valve and tap the syringe lightly to detach any trapped air bubbles. Open the valve and expel the air bubbles, leaving between 50 and 60 mL of sample in the syringe. Do not leave more than 60 mL of sample in the syringe. Close the valve.

- 8. Fill two syringes (pH and DIC) following steps 1-7 above and place the syringes together in a sealable plastic bag in the syringe protective case in the cooler.
- 9. Fill a second set of two syringes (aluminum analyses) following steps 1-7 above and place the syringes together in a second sealable plastic bag in the same syringe protective case in the cooler.

### 6.4.6 Hydrologic Measurements

All hydrologic data should be recorded on a form similar to the NSWS Hydrologic Data Form (Appendix A, Figure A-9). Hydrologic measurements are taken only at the downstream site for each stream. Sampling personnel should enter a stream only if they can do so safely. Section 3.8.2 presents appropriate safety considerations. Figure 6-1 depicts the course of action for hydrology measurements.

## 6.4.6.1 Electromagnetic Current Meter Calibration Check--

NOTE: This procedure has been written assuming a Marsh-McBirney Model 201D electromagnetic current meter is being used. This procedure may be used, with modification, with other meters meeting equivalent specifications.

- 1. Meter calibration should be checked daily during routine morning calibration and again onsite before entering the stream.
- 2. The value should be  $10.00 \pm 0.20$ . The value obtained during morning calibration should be recorded in the comments section of the calibration form. The values obtained onsite should be recorded on the Hydrologic Data form.
- 3. Once a week the zero value should be checked in static water. The probe should sit for 30 minutes with no disturbance. The value obtained should be 0.0  $\pm$  0.1. The meter zero should be adjusted if it is outside this range.

### 6.4.6.2 Stream Stage--

- 1. On the first visit to each downstream site, a steel rod should be hammered into the streambed at a location which is out of the main flow, protected from debris, and not affected by bilateral flows from another stream.
- 2. Stream stage should be measured relative to the top of the rod twice during the first visit, once immediately upon placement and again just prior to leaving the site following sampling. Whenever possible, the elevation of the top of the steel rod (the reference point) will be considered to be 3.00 feet, and stream stage measurements will be relative to this value.
- 3. If an existing gauging station is available, it should be used, in addition to the steel rod, for all gauge measurements at this site.

### 6.4.6.3 Discharge Measurement--

On each visit, upon arrival at the downstream site, again measure stream stage relative to the top of the steel rod. Measure stream discharge at each downstream site as described below:

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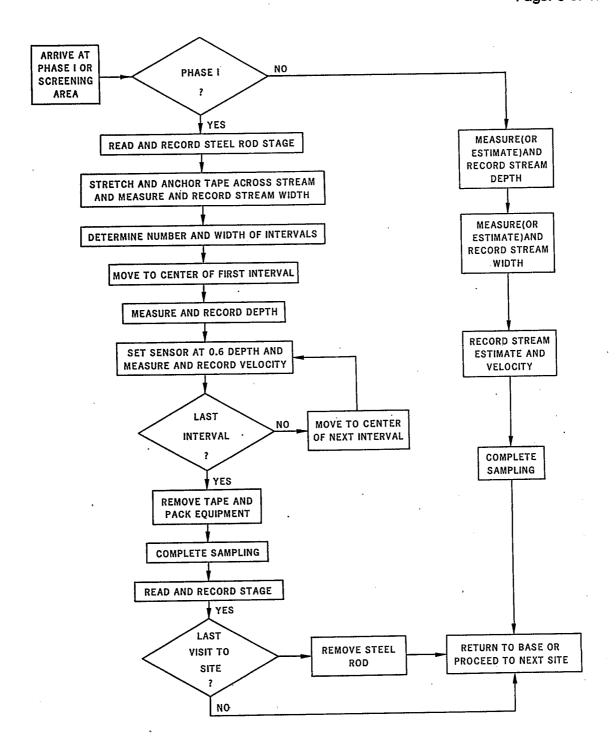


Figure 6-1. Flowchart for hydrology measurements.

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- Facing downstream and beginning on the right edge of the water (REW), stretch a meter tape across the stream perpendicular to stream flow at a uniform section of the channel.
   If possible the channel should be approximately U-shaped, with no eddies or turbulence.
- 2. Measure and record the stream width. Leave the tape tightly suspended across the stream, approximately one foot above water level.
- 3. Divide the total stream width into approximately ten equal-sized intervals. The minimum number of intervals should be eight; the maximum number should be fifteen. To determine interval width, divide the total stream width by an integer value near ten and then round down to a convenient number. An additional interval should be added if this procedure results in an unmeasured section of stream greater than or close to one interval in width.
- 4. Attach an electromagnetic current meter probe (Marsh-McBirney Model 201D or equivalent) to a wading rod and check the internal electronics by turning the switch to "CAL." If the meter calibrates in air, proceed with the measurements. The calibration check reading is recorded in the comments section of the Hydrologic Data form (10.00 ± 0.20).
- 5. Move to the center of the first interval from the REW.
- 6. Read and record the stream depth at the center of the interval.
- 7. Place the current meter probe at 0.6 of the total depth (measured from surface) or 0.4 of the total depth as measured from the bottom. Orient the probe properly in the flow. Wait 20 seconds to allow the meter to equilibrate, then measure and record the current velocity at the center of the interval. Use the lowest time constant scale that provides stable readings.
- 8. Repeat steps (6) and (7) for all intervals.
  - NOTE: Interval depth is measured to  $\pm$  0.05 ft and stage is measured to  $\pm$  0.01 ft. All other measurements are in metric units.
- 9. When sampling is completed and just prior to departure, again measure stage height by using the steel rod.
- 10. Remove the steel rod from the site.

### 6.4.6.3 Hazardous Stream Conditions--

If conditions are too dangerous to enter the stream, use techniques described in this section to estimate stream discharge.

- 1. Base an estimate of stream discharge on measurements or estimates of stream width, mean channel depth, and mean current velocity.
- 2. Measure stream width with a meter tape or by the following method:
  - a. Facing the other shore, stand at the edge of the stream on land that is at the same elevation as the water surface.

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- b. Sight down the length of one extended arm toward the other shore.
- c. Holding the extended arm at a fixed angle from the horizontal, pivot around until the arm is pointing toward a location your partner can easily mark and which is at the same elevation as the water surface.
- d. Measure the distance from your feet to the mark and record this distance on the data form as the estimated stream width.
- 3. Estimate mean channel depth by the following technique:
  - a. Estimate and record the mean depth of the whole channel area over which velocity estimates will be made.
  - b. If there is more than one stream channel, record the mean depth and the width of each one and note this information in the comments section of the Hydrologic Data form.
  - c. If the stream bottom is visible, sketch a cross section of the channel on the back of the field Hydrologic Data form.
- 4. Estimate current velocity by the following technique:
  - a. Choose a section of stream that is relatively straight and free of obstructions.
  - b. Measure and mark a distance of 2 to 10 meters along the shoreline, depending on the size of the stream.
  - c. Drop an apple or an orange into the stream upstream of the starting point.
  - d. Measure the amount of time required for the object to be carried through the measured section.
  - e. Divide the measured distance by the measured amount of time to obtain an estimate of velocity (± 0.1 m/sec).
  - f. Repeat (c), (d), and (e) two more times. Record the average value of the three trials on the data form and mark on the data form that flow was estimated, not measured.

### 6.5 Safety

All sampling personnel should be fully trained and competent in all skills outlined in this section and must fully understand all safety procedures discussed. While away from the base area, team members are responsible for their own safety and for each other's safety. General field safety considerations are discussed in Section 3.8.2. Specialized training for stream sampling includes wilderness survival and orienteering.

### 6.5.1 Wilderness Travel and Camping

On many occasions several miles of hiking with heavy packs may be necessary to reach sites. Samplers must be competent in wilderness survival skills in order to be fully prepared to handle all conditions and situations that may arise.

Topics covered in survival training should include thermoregulation, methods of heat exchange, wet versus dry cold, physical response to cold, hypothermia, frostbite, and insulation qualities of clothing types. In addition, poisonous plants, dangerous animals, and insects likely to be encountered during sampling operations should be discussed.

### 6.5.2 Map Reading, Compass Use, and Orienteering

Samplers must be competent at map reading, compass use, and orienteering. They will be required to determine and mark on a topographic map the exact location at which streams were sampled; use maps, landmarks, and compasses to locate and travel to stream sites where no trails exist; and determine the orientation of streams. Competency in these skills is essential for safe wilderness travel.

A full course in map and compass use, including a field orienteering practical skills session, should be taught.

### 6.5.3 Sampling in Flowing Water

- 1. Samplers should receive a training course in stream crossing and belaying, tetherline use, and in-stream rescue.
- 2. Samplers should be supplied with chest waders or hip waders for use while sampling.
- 3. A safety line is recommended when entering water over 2 feet cleep, streams where footing is unsure, streams with rapidly flowing water, or when working in streams at night. Flowing water over 3 feet deep or streams with extremely slippery streambeds should not be entered.
- 4. Samplers should not enter a stream if they are alone at the site. When crossing streams, no more than one sampler should be in the water at one time.
- 5. When entering water at night or in poor light, samplers should exercise extreme caution in selecting foot placements and in movement. A headlamp should be used to allow freedom of hand movement for balance and for handling instruments.

# 7.0 Determination of pH, Specific Conductance, Dissolved Oxygen, and Temperature Using an Integrated Monitoring System

### 7.1 Overview

### 7.1.1 Scope and Application

This method uses one integrated monitoring system to measure pH, specific conductance, dissolved oxygen (DO), and temperature in low ionic strength waters. An integrated monitoring system is advantageous (as compared to the use of separate meters) for several reasons. Only a single sonde (underwater unit) and cable are needed, and several measurements can be made simultaneously. This procedure is a compilation of similar procedures utilized during the AERP lake surveys.

Measurements of pH, specific conductance, DO, and temperature are made at selected intervals throughout the water column. For this reason, an extended cable is required. The AERP surveys relied on Hydrolab model 4041 and Hydrolab Surveyor II sondes and meters. Both units function in a similar manner with only minor differences in calibration techniques. The method described here assumes that the Hydrolab Surveyor II and sonde are used. The method can be modified and used for other instrumentation meeting equivalent specifications.

The basic system consists of five components: a display unit, data cable, sonde, circulator, and battery pack (Figure 7-1). The Hydrolab pH system used in the AERP was modified with a Beckman Red Label Lazaran reference electrode and a Beckman glass measuring electrode to provide greater sensitivity in low ionic strength waters. Hydrolabs are used primarily for lakes rather than streams; they are used to establish temperature stratification profiles and to determine certain chemical characteristics.

# 7.1.2 Summary of Method

When the Hydrolab is in operation, all parameters are measured simultaneously at the sonde unit. The resulting signals are transmitted in parallel up the cable to the display unit. In the display unit, the signals may be amplified or shifted. After this processing, the signals are ready to be selected by the user (via the panel switch) for digital conversion and immediate display.

Calibration controls for each measurement are provided on the front panel of the display unit. These controls are used to adjust the instrument before going to the field.

Hydrolabs should be calibrated before in situ measurements are taken. Calibration settings should be checked using a QCC solution immediately after calibration and again after field measurements have been made. A field QCC should also be done on site prior to in situ measurement. Daily and weekly maintenance procedures established by the manufacturer should be followed.

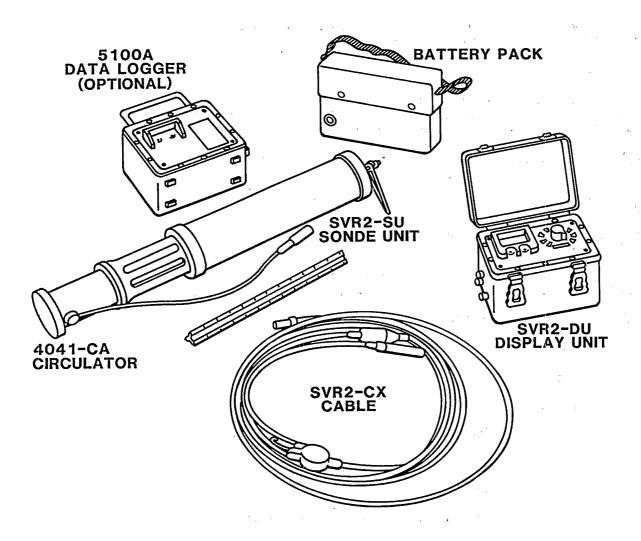


Figure 7-1. Hydrolab system components.

#### 7.1.3 Interferences

The instrument should be at thermal equilibrium during calibration with the solutions being measured and when in situ measurments are taken. Temperature change affects instrument calibration and stability. If possible, the Hydrolab should be kept at temperatures above -10 °C. Store the Hydrolab and calibration solutions in the same area.

Sonde sensor function is degraded gradually by immersion in natural waters containing oils, plankton, and colloids. These cause a film to form on the sonde. Routine maintenance procedures must be followed to keep sensors free of such film. Contact with sediments will degrade sensor

function. When sampling in lakes, do not allow the sonde to drop into sediments. Low ionic strength waters may require long equilibration times.

#### 7.1.4 Safety

The calibration standards and protocols in this method pose no hazard to the sampler. General safety guidelines for samplers operating on lakes and under remote conditions are provided in sections 3.8, 4.5, and 5.5. Additional helicopter safety guidelines are contained in Appendix B.

# 7.2 Sample Collection, Preservation, and Storage

Because lake chemistry measurements are determined in situ; sample collection, preservation, and storage are not applicable.

# 7.3 Equipment and Supplies

A Hydrolab Surveyor II system or its equivalent is required for lake chemistry measurements. Supplies and other materials are described in sections 7.3.1 - 7.3.3.

### 7.3.1 Apparatus and Materials

- 1. Surveyor II manual.
- 2. Calibration cup and soft rubber cap.
- 3. Spare storage cup with hard white cover.
- 4. Plastic bucket (for discarded solutions).
- 5. Calibration stand (ringstand and vise clamp).
- 6. NBS-traceable thermometer.
- 7. Barometer, altimeter, or phone number of local weather bureau. Alternately, correct for elevation.
- 8. 3 M KCl electrolyte solution.
- Battery chargers.

#### 7.3.2 Consumable Materials

- 1. Standardized calibration and field forms.
- 2. Soft paper wipes (Kimwipes or equivalent).
- 3. Complete maintenance kit, including cotton swabs (Q-tips), DO sensor papers, DO electrolyte solution, small scissors, emory paper, silicone grease, small screwdriver, and isopropyl alcohol.

4. CRC (ether) spray.

#### 7.3.3 Reagents

Water--Water used in all preparations should conform to American Society for Testing and Materials (ASTM) specifications for Type I reagent grade water (ASTM, 1984).

pH calibration buffers--National Bureau of Standards (NBS)-traceable pH buffers at pH 4.00 and 7.00 (at 25 °C).

### 7.3.4.1 Potassium Chloride Stock Solution (1 N KCI)--

This stock solution is used to make the 147  $\mu$ S/cm standard. It should be prepared in at least 1-L batches to minimize weighing and dilution errors. Prepare as needed and refrigerate at 4 °C. The 1 N KCl stock solution has a theoretical specific conductance of 111,900  $\mu$ S/cm at 25 °C. This value should be verified by measuring at least three 35-mL samples contained in 50-mL centrifuge tubes.

- 1. Fill a clean 1-L volumetric flask with approximately 500 mL of deionized water.
- 2. Weigh 74.553 g of potassium chloride (KCl, ultrapure, dried for 2 hours at 105 °C and ampulated).
- 3. Completely dissolve the KCI in deionized water and dilute to the 1-L mark. Mix again thoroughly.
- 4. Store the stock solution in 500-mL bottles (deionized water-washed) that have been rinsed three times with the 1N KCI solution. Label the bottles "1 N KCI Stock Solution" and refrigerate at 4 °C.

# 7.3.4.2 Specific Conductance QCC Solution (0.001 N KCl)--

- Fill a clean, labeled 1-L volumetric flask with approximately 500 mL of deionized water. Obtain a 50-mL disposable beaker, rinse three times with 1 N KCI stock solution, and pour 5 to 10 mL of stock solution into the beaker.
- 2. Use a calibrated 100- to 2,000-μL pipet (rinse pipet tip one time with solution) to deliver 1.000 mL of stock solution to the 1-L flask. Mix and dilute to the 1-L mark and mix again.
- 3. Label the containers and refrigerate the solutions if possible. If the solutions cannot be refrigerated, store them in a cool, dark location.

# 7.3.4.3 pH 4.00 QCC Solution--

- 1. Prepare daily if possible.
- 2. Add 1.0 mL of 0.1 N H<sub>2</sub>SO<sub>4</sub> to a clean, 1-L volumetric flask; dilute acid to 1 L with deionized water.

# 7.4 Preparation

### 7.4.1 Instrument Assembly

- 1. Apply an extremely thin film of silicone grease to all soft black rubber connections to provide watertight seals. Do not allow silicone grease to contact the pin connectors. Line up the raised dot on the 4-conductor socket of the data cable with the large pin at the top of the sonde housing. Connect the 4-conductor socket of the data cable with the large pin at the top of the sonde housing.
- 2. Connect the metal bail on the sonde to the eye screw on the data cable via the toggle, clevis pin, and pin retainer.
- Attach the metal locking connector at the surface end of the data cable to the labeled TRANSMITTER socket of the display unit. Line up the keys and grooves, slide the plug into the socket, and rotate the knurled locking ring to the right until it clicks.
- 4. Connect the battery pack to the display unit via the battery pack cable. Verify that the display unit FUNCTION switch is OFF. Attach the end of the cable to the labeled 12 VOLTS DC socket on the display unit and lock it into position.
- 5. Remove the storage cup from the end of the sonde.
- 6. Verify that all connections have been made and tightened. Switch FUNCTION to BATT position. The acceptable operating range is 11.5 to 13.9 volts. Replace the battery pack if the voltage is less than 11.5.
- 7. Switch the display unit to TEMP and verify that the display unit initiates a high speed self-test for approximately five seconds before displaying the temperature. If an error message appears, consult the error message listing on the display unit lid and the trouble-shooting section of the Hydrolab user's manual.

# 7.4.2 Hydrolab Circulator Assembly and Test

The circulator assembly is required for measurement of DO in static waters and as a housing to protect the fragile sensors located at the tip of the Hydrolab sonde unit. To attach the circulator and check its operation proceed as follows:

- 1. Screw on the circulator.
- 2. Connect the 2-conductor socket of the data cable to the circulator. The two pins are different sizes; it is critical to mate them properly and to use a straight motion to prevent damage to the connector pins. Use silicone grease on the rubber connections.
- Switch the display unit on and verify that the circulator motor starts and the impeller rotates freely.

NOTE: The circulator is attached just before field measurements are taken; it is not to be attached during calibration, except to confirm proper operation.

## 7.4.3 Preparation for Calibration

- 1. Mount vise clamp to ringstand. Remove storage cup from sonde.
- 2. Remove KCl storage cap (soft black rubber cap) from pH reference probe.

NOTE: Cap may be covered with Parafilm to prevent loss of KCl solution. Do not replace KCl cap until post-field QCC has been performed.

3. Screw calibration cup on the sonde unit. Set FUNCTION switch to TEMP. Once turned on, the display unit must not be turned off until all calibrations are completed and saved.

#### 7.4.4 Rinse Procedure

Before and after each sensor calibration, rinse the sensor as follows:

- 1. Fill calibration cup one-third full with deionized water.
- 2. Snap on soft cover and shake sonde for 10 seconds, contacting all surfaces with deionized water.
- 3. Pour out water. Repeat twice more using fresh deionized water.
- 4. Remove cap and shake off excess water from sensors.

# 7.5 Hydrolab Calibration

The Hydrolab should be calibrated in the morning of each sampling day. Specific conductance should be standardized with a 0.001 N KCl solution (specific conductance = 147  $\mu$ S/cm at 25 °C). To standardize the pH electrode, NBS-traceable buffers (pH = 4.00 and pH = 7.00 at 25 °C) should be used. Dissolved oxygen measurements are calibrated with water-saturated air. This procedure must be performed in a temperature-controlled environment to ensure thermal equilibrium of the solutions.

Following acceptable calibration, the calibration should be checked using a QCC solution, a standard of low ionic strength (0.001 N sulfuric acid solution) for pH (4.03 at 25 °C) and specific conductance (42  $\mu$ S/cm at 25 °C) measurements. If measurements of the QCC solution differ from the theoretical values by more than 0.20 pH unit or by more than 15  $\mu$ S/cm, then the Hydrolab must be recalibrated. If the recalibration fails, maintenance procedures should be performed. The Hydrolab temperature probe should be checked by comparing the temperature reading of the QCC solution to that from an NBS-traceable thermometer. The Hydrolab temperature reading should be within ±1 °C of the NBS-traceable thermometer reading. Spare Hydrolabs should be available in the field.

## 7.5.1 pH Calibration

The applicable pH range is between 3.0 and 8.0 pH units. The range may be extended by the use of a wider range of pH calibration standards and pH quality control check (QCC) solutions.

The following steps must be completed for pH calibration:

- 1. Complete rinse procedure (Section 7.4.4).
- 2. Rinse three times with small quantities of pH 7 buffer solution and discard.
- 3. Fill calibration cup with stock pH 7 buffer solution to a level just above the DO membrane and mount sonde on ringstand.
- 4. Allow three minutes for the sonde and buffer solution to reach thermal equilibrium. Monitor on TEMP display for stabilization.
- 5. Determine buffer pH in relation to buffer temperature (Table 7-1). Switch display to pH. Use the ZERO toggle switch to adjust display pH to the value near 7.00 which is determined from Table 7-1.

Table 7-1. Temperature Correction Factors for pH Buffers<sup>a</sup>

Temperature	But	fers	
°C	pH 4.00	pH 7.00	
0	4.01	7.12	
5	4.01	7.09	
10	4.00	7.06	
15	4.00	7.04	
20	4.00	7.02	
25	4.00	7.00	
30	4.01	6.99	
35	4.02	- 6.98	
40	4.03	6.98	
45	4.04	6.97	
50	4.06	6.97	

<sup>&</sup>lt;sup>a</sup> Values given are for pH 4 reference buffer solution (National Bureau of Standards-Traceable, SRM 185e) and pH 7 reference buffer solution (National Bureau of Standards-Traceable, SRM 186-1 and 186-11-c), prepared by American Scientific Products, McGaw Park, IL, 60085.

- 6. Repeat steps 1 through 4 using stock pH 4 buffer solution, instead of pH 7.
- 7. Switch display to pH. Use the SLOPE toggle switch to adjust the displayed value to the correct buffer pH value as determined from Table 7-1.
- 8. Repeat steps 1-4 a second time for pH 7. If the value displayed is outside the acceptable range, the entire pH calibration procedure must be repeated.
- 9. After pH sensors have been calibrated to within acceptable limits, save the calibration (see Section 7.5.4).

# 7.5.2 Specific Conductance Calibration

The Surveyor II has three specific conductance ranges and automatically switches to the appropriate range for the values being measured. By calibrating with a standard that is above,

but as near as possible to the expected data range, the most precise data will be obtained. The following steps must be completed:

- 1. Complete the rinse procedure (Section 7.4.4). Fill the calibration cup about one-third full with 147  $\mu$ S/cm standard solution.
- 2. Cover the cup and shake the sonde; discard solution. Repeat a second and third time.
- 3. Mount the sonde in a vise clamp; fill the calibration cup with standard solution to a level above the specific conductance block. The bores of the sensor must not contain any air bubbles. If any bubbles are present, tap the calibration cup lightly to dislodge the bubbles, or refill the calibration cup.
- 4. Allow 1 to 3 minutes for the sonde and the standard solution to reach thermal equilibration.
- 5. Switch to TEMP; verify that the reading is stable.
- 6. Switch to COND. Use the SLOPE toggle switch to adjust the displayed reading to the standard value (147  $\mu$ S/cm).
- 7. If the instrument cannot be adjusted to 147  $\mu$ S/cm, recalibrate it with fresh standard. If the problem persists, perform routine maintenance.
  - NOTE: The instrument should be calibrated with the 147  $\mu$ S/cm solution at room temperature. If the solution is below room temperature, calibrating to 147  $\mu$ S/cm may be difficult.
- 8. After specific conductance sensors have been accurately calibrated, save the calibration (Section 7.5.4).

# 7.5.3 Dissolved Oxygen Calibration

Before beginning the DO calibration, verify that the DO membrane is in good condition (Section 7.8.2). The standard for the DO calibration is water-saturated air; temperature and barometric pressure affect the value of this standard. The sonde provides a temperature measurement; absolute barometric pressure may be obtained from a mercury barometer, local airport, or weather bureau.

NOTE: Be sure the barometric pressure is not corrected to sea level. If it is, it can be uncorrected by using the following formula:

Uncorrected BP = Corrected BP ≥ 2.5 (A/100)

BP: Barometric pressure

A: Local altitude above sea level (feet)

DO calibration involves the following steps:

1. After rinsing (Section 7.4.4), fill the calibration cup with deionized water so that the DO membrane is submerged about 1 cm. The cup will be nearly full.

- 2. Snap on soft cover and agitate sonde gently for about 15 seconds. Set FUNCTION to TEMP. Remove soft cover. Monitor readings; if temperature changes more than 0.1 °C in 5 seconds, replace cover on sonde and repeat agitation.
- 3. When a stable temperature has been achieved, remove cover and carefully pour off enough water so that the membrane is about 0.5 cm above the liquid. Blot away any water droplets on the membrane surface with a Kimwipe or cotton swab.
- 4. Place the storage cap upside-down on top of the calibration cup. This is to keep air currents out of the cup without changing the pressure in the cup. Wait 5 minutes.
- 5. Read the temperature; consult Table 7-2 for the DO concentration corresponding to that temperature and the local absolute barometric pressure. Record this value. Use the SLOPE toggle switch to set the display to the recorded value.

NOTE: Move the toggle switch toward the display to increase the value of the reading; move the switch away from the display to decrease the value.

6. Save calibration (Section 7.5.4).

### 7.5.4 Saving Calibration

After completing each sensor calibration, save calibration as follows:

- 1. Switch display to BATT.
- 2. Pull both calibration toggle switches simultaneously toward you.
- 3. Wait until SAVE appears in the display, then release the switches.

NOTE: Do not turn the instrument OFF until all sensors have been calibrated and saved, or sensor calibrations will be lost.

#### 7.6 Procedure

#### 7.6.1 Premeasurement Procedure

Initial calibration (Section 7.5) and the calibration QCC (Section 7.7.1) should be performed in a controlled-temperature environment prior to transporting the Hydrolab instrument to the field.

The system should be kept intact during transport. If it is necessary to disassemble the instrument, dust caps should be installed on all connectors and sockets to prevent moisture from entering. The system should be protected from vibration and extreme temperature. Probes must not dry out. They should be stored in tap water or temporarily in lake water, but not in deionized water.

Complete the field QCC (Section 7.7.2) in an area protected from the wind, direct sunlight, and other disturbances.

Table 7-2. Oxygen Solubility at Indicated Pressure

emperature	Pressure (mm Hg)								
*c	P(H <sub>2</sub> 0)	760	755	750	745	740	735	730	
0	4.58	14.57	14.47	14.38	14.28	14.18	14.09	13.99	
0 1 2 3 4 5 6 7 8 9	4.93	14.17	4.08	13.98	13.89	13.79	13.70	13.61	
ż	5.29	13.79	13.70	13.61	13.52	13.42	13.33	13.24	
3	5.68	13.43	13.34	13.25	13.16	13.07	12.98	12.90	
4	6.10	13.08	12.99	12.91	12.82	12.73	12.65	12.56	
5	6.54	12.74	12.66	12.57	12.49	12.40	12.32	12.23	
6	7.01	12.42	12.34	12.26	12.17	12.09	12.01	11.93	
7	7.51	12.11	12.03	11.95	11.87	11.79	11.71	11.63	
8	8.04	11.81	11.73	11.65	11.57	11.50	11.42	11.34	
9	8.61	11.53	11.45	11.38	11.30	11.22	11.15	11.07	
10	9.21	11.26	11.19	11.11	11.04	10.96	10.89	10.81	
11	9.84	10.99	10.92	10.84	10.77	10.70	10.62	10.55	
12	10.52	10.74	10.67	10.60	10.53	10.45	10.38	10.31	
13	11.23	10.50	10.43	10.36	10.29	10.22	10.15	10.08	
14	11.99	10.27	10.20	10.13	10.06	10.00	9.93	9.86	
15	12.79	10.05	9.98	9.92	9.85	9.78	9.71	9.65	
16	13.63	9.83	9.76	9.70	9.63	9.57	9.50	9.43	
17	14.53	9.63	9.57	9.50	9.44	9.37	9.31	9.24	
18	15.48	9.43	9.37	9.30	9.24	9.18	9.11	9.05	
19	16.48	9.24	9.18	9.12	9.05	8.99	8.93	8.87	
20	17.54	9.06	9.00	8.94	8.88	8.82	8.75	8.69	
21 22	18.65	8.88	8.82	8.76	8.70	8.64	8.58	8.52	
22	19.83	8.71	8.65	8.59	8.53	8.47	8.42	8.36	
23	21.07	8.55	8.49	8.43	8.38	8.32	8.26	8.20	
24	22.38	8.39	8.33	8.28	8.22	8.16	8.11	8.05	
25	23.76	8.24	8.18	8.13	8.07	8.02	7.96	7.90	
26	25.21	8.09	8.03	7.98	7.92	7.87	7.81	7.70	
27 28	26.74	7.95	7.90	7.84	7.79	7.73	· 7.68	7.62	
28	28.35	7.81	7.76	7.70	7.65	7.60	7.54	7.49	
29	30.04	7.68	7.63	7.57	7.52	7.47	7.42	7.36	
30	31.82	7.55	7.50	7.45	7.39	7.34	7.2 <del>9</del>	7.24	
31	33.70	7.42	7.37	7.32	7.27	7.22	7.16	7.11	
32	35.66	7.30	7.25	7.20	7.15	7.10	7.05	7.00	
33	37.73	7.08	, <b>7.13</b>	7.08	7.03	6.98	6.93	6.88	
34	39.90	7.07	7.02	6.97	6.92	6.87	6.82	6.78	
35	42.18	6.95	6.90	6.85	6.80	6.76	6.71	6.66	
36	44.56	6.84	6.79	6.76	6.70	6.65	6.60	6.5	
37	47.07	6.73	6.68	6.64	6.59	6.54	6.49	6.4	
38	49.69	6.63	6.58	6.54	6.49	6.44	6.40	6.3	
39	52.44	6.52	6.47	6.43	6.38	6.35	6.29	6.24	
40	55.32	6.42	6.37	6.33	6.28	6.24	6.19	6.15	
41	58.34	6.32	6.27	6.23	6.18	6.14	6.09	6.05	
42	61.50	6.22	6.18	6.13	6.09	6.04	6.00	5.95	
43	64.80	6.13	6.09	6.04	6.00	5.95	5.91	5.87	
44	68.26	6.03	5.99	5.94	5.90	5.86	5.81	5.77	
45	71.88	5.94	5.90	5.85	5.81	5.77	5.72	5.68	

(continued)

Table 7-2. Continued

Temperature	Pressure (mm Hg)								
•c	725	720	715	710	705	700	695	690	
o o	13.89	13.80	13.70	13.61	13.51	13.41	13.32	13.22	
ĭ	13.51	13.42	13.33	13.23	13.14	13.04	12.95	12.86	
2	13.15	13.06	12.07	12.88	12.79	12.69	12.60	12.51	
3	12.81	12.72	12.63	12.54	12.45	12.36	12.27	12.18	
4	12.47	12.39	12.30	12.21	12.13	12.04	11.95	11.87	
5	12.15	12.06	11.98	11.89	11.81	11.73	11.64	11.56	
6	11.84	11.73	11.68	11.60	11.51	11.43	11.35	11.27	
7	11.55	11.47	11.39	11.31	11.22	11.14	11.06	10.98	
8	11.26	11.18	11.10	11.02	10.95	10.87	10.79		
.9	10.99	10.92	10.84	10.76			10.79	10.71	
10	10.99	10.66	10.59	10.76	10.69 10.44	10.61 10.36	10.53 10.29	10.46	
11	10.48	10.40	10.33	10.26	10.44	10.36		10.21	
12	10.46	10.40	10.10	10.28	9.95	9.88	10.04	9.96	
13	10.24	9.94	9.87	9.80			9.81	9.46	
13				9.60	9.73	9.66	9.59	9.52	
14	9.79	9.72	9.65	9.68	9.51	9.45	9.38	9.31	
15	9.58	9.51	9.44	9.58	9.31	9.24	9.18	9.11	
16	9.37	9.30	9.24	9.17	9.11	9.04	8.97	8.91	
17	9.18	9.11	9.05	8.98	8.92	8.85	8.79	8.73	
18	8.99	8.92	8.86	8.80	8.73	8.67	8.61	8.54	
19	8.81	8.74	8.68	8.62	8.56	8.49	8.43	8.37	
20	8.63	8.57	8.51	8.45	8.39	8.33	8.27	8.21	
21	8.46	8.40	8.34	8.28	8.22	8.16	8.10	8.04	
22	8.30	8.24	8.18	8.12	8.06	8.00	7.95	7.89	
23 24	8.15	8.09	8.03	7.97	7.91	7.86	7.80	7.74	
2 <del>4</del> 25	7.99 7.85	7.94 7.79	7.88 7.74	7.82	7.76	7.71	7.65	7.59	
26 26	7.00		7.74	7.68	7.60	7.57	7.51	7.46	
26 27	7.70 7.57	7.65 7.52	7.59	7.54	7.48	7.43	7.37	7.32	
27 28	7.37 7.44		7.46	7.41	7.35	7.30	7.25	7.19	
26 29		7.38	7.33	7.28	7.22	7.17	7.12	7.06	
29 30	7.31 7.19	7.26 7.14	7.21	7.15	7.10	7.05	7.00	6.94	
30			7.08	7.03	6.98	6.93	6.88	6.82	
31 · 32	7.06	7.01	6.96	6.91	6.86	6.81	6.76	6.70	
3 <u>2</u>	6.95	6.90	6.85	6.80	6.70	6.70	6.64	6.59	
33 34	6.83	6.78	6.73	6.68	6.63	6.58	6.53	6.48	
3 <del>4</del> 35	6.73 6.61	6.68	6.63	6.58	6.53	6.48	6.43	6.38	
35 36		6.56	6.51	6.47	6.42	6.37	6.36	6.27	
36 37	6.51	6.46	6.41	6.36	6.31	6.27	6.22	6.17	
37 38	6.40	6.35	6.31	6.26	6.21	6.16	6.12	6.07	
38 39	6.30 6.26	6.26	6.21	6.16	6.12	6.07	6.02	5.98	
		6.15	6.11	6.06	6.01	5.97	5.92	5.87	
40	6.10	6.06	6.01	5.96	5.92	5.86	5.83	5.78	
41	6.00	5.96	5.91	5.87	5.82	5.78	5.73	5.69	
42	5.91	5.86	5.82	5.77	5.73	5.69	5.64	5.60	
43	5.82	5.78	5.73	5.69	5.65	5.60	5.56	5.51	
44	5.72	5.68	5.64	5.59	5.55	5.51	5.46	5.42	
45	5.64	5.59	5.55	5.51	5.47	5.42	5.38	5.34	

(continued)

Table 7-2. Continued

Temperature	Pressure (mm Hg)								
*c	685	680	675	670	665	660	655	650	
0	13.12	13.03	12.93	12.83	12.74	12.64	12.54	12.45	
	12.76	12.67	12.57	12.48	12.39	12.29	12.20	12.11	
1 2 3 4 5 6 7 8	12.42	12.33	12.24	12.15	12.05	11.96	11.87	11.78	
3	12.09	12.01	11.92	11.83	11.74	11.65	11.56	11.47	
4	11.78	11.69	11.61	11.52	11.43	11.35	11.26	11.17	
5	11.47	11.39	11.30	11.22	11.13	11.05	10.96	10.88	
ě	11.18	11.10	11.02	10.94	10.85	10.77	10.69	10.61	
7	10.90	10.82	10.74	10.66	10.58	10.50	10.42	10.34	
ġ	10.63	10.55	10.48	10.40	10.32	10.24	10.16	10.08	
9	10.38	10.30	10.23	10.15	10.07	10.00	9.92	9.84	
10	10.14	10.06	9.99	9.91	9.84	9.76	9.69	9.61	
11	9.89	9.82	9.74	9.67	9.60	9.52	9.45	9.38	
12	9.67	9.59	9.52	9.45	9.38	9.31	9.24	9.16	
13	9.45	9.38	9.31	9.24	9.17	9.10	9.03	8.96	
14	9.24	9.17	9.10	9.03	8.97	8.90	8.83	8.79	
15	9.04	8.97	8.91	8.84	8.77	8.70	8.64	8.57	
16	8.84	8.78	8.71	8.64	8.58	8.51	8.45	8.38	
17	8.66	8.60	8.53	8.47	8.40	8.34	8.27	8.21	
18	8.48	8.42	8.35	8.29	8.23	8.16	8.10	8.04	
19	8.31	8.25	8.18	8.12	8.06	8.00	7.94	7.87	
20	8.14	8.08	8.02	7.96	7.90	7.84	7.78	7.72	
21	7.98	7.92	7.86	7.80	7.74	7.68	7.62	7.56	
22	7.83	7.77	7.71	7.65	7.59	7.53	7.47	7.42	
23	7.68	7.62	7.57	7.51	7.45	7.39	7.34	7.28	
24	7.54	7.48	7.42	7.37	7.31	7.25	7.20	7.14	
25	7.40	7.34	7.29	7.23	7.18	7.12	7.06	7.0	
26	7.26	7.21	7.15	7.10	7.04	6.99	6.93	6.8	
27	7.14	7.08	7.03	6.97	6.92	6.87	6.81	6.70	
28	7.01	6.96	6.90	6.85	6.80	6.74	6.69	6.6	
29	6.89	6.84	6.79	6.73	6.68	6.63	6.58	6.5	
30	6.77	6.72	6.67	6.62	6.57	6.51	6.46	6.4	
31	6.65	6.60	6.55	6.50	6.45	6.40	6.35	6.3	
32	6.54	6.49	6.44	6.39	6.34	6.29	6.24	6.19	
33	6.43	6.38	6.34	6.29	6.24	6.19	6.14	6.0	
34	6.33	6.28	6.24	6.19	6.14	6.09	6.04	5.9	
35	6.22	6.18	6.13	6.08	6.03	5.98	5.93	5.8	
36	6.12	6.08	6.03	5.98	5.93	5.88	5.84	5.7	
37	6.02	5.97	5.93	5.88	5.83	5.79	5.74	5.6	
38	5.93	5.88	5.84	5.79	5.74	5.70	5.65	5.6	
39	5.83	5.78	5.74	5.69	5.64	5.60	5.55	5.5	
40	5.74	5.69	5.65	5.60	5.55	5.51	5.45	5.4	
41	5.64	5.60	5.55	5.51	5.46	5.42	5.37	5.3	
42	5.55	5.51	5.46	5.42	5.37	5,33	5.28	5.2	
43	5.47	5.42	5.38	5.34	5.29	5.25	5.20	5.1	
44	5.38	5.33	5.29	5.25	5.20	5.16	5.11	5.0	
45	5.29	5.25	5.21	5.16	5.12	5.08	5.03	4.9	

(continued)

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Table 7-2. Continued

Temperature	Pressure (mm Hg)								
•c	645	640	635	630	625	620	615	610	
0	12.35	12.26	12.16	12.06	11.97	11.87	11.77	11.68	
0 1 2 3 4 5 6 7 8	12.01	11.92	11.82	11.73	11.64	11.54	11.45	11.36	
2	11.69	11.60	11.51	11.41	11.32	11.23	11.14	11.05	
3	11.38	11.29	11.20	11.12	11.03	10.94	10.85	10.76	
4	11.08	11.00	10.91	10.82	10.74	10.65	10.56	10.48	
5	10.80	10.71	10.63	10.54	10.46	10.37	10.29	10.20	
6	10.52	10.44	10.36	10.28	10.19	10.11	10.03	9.95	
7	10.26	10.18	10.10	10.02	9.94	8.86	9.78	9.70	
8	10.00	9.93	9.85	9.77	9.69	9.61	9.53	9.45	
9	9.77	9.69	9.61	9.54	9.46	9.38	9.30	9.23	
10	9.54	9.46	9.39	9.31	9.24	9.16	9.09	9.01	
11	9.31	9.23	9.16	9.09	9.01	8.94	8.87	8.79	
12	9.09	9.02	8.95	8.88	8.81	8.73	8.66	8.59	
13	8.89	8.82	8.75	8.68	8.61	8.54	8.47	8.40	
14	8.69	8.62	8.55	8.49	8.42	8.35	8.28	8.21	
15	8.50	8.44	8.37	8.30	8.23	8.17	8.10	8.03	
16	8.32	8.25	8.18	8.12	8.05	7.99	7.92	7.85	
17	8.14	8.08	8.02	7.95	7.89	7.82	7.74	7.69	
18	7.97	7.91	7.85	7.78	7.72	7.66	7.59	7.53	
19	7.81	7.75	7.69	7.62	7.56	7.50	7.44	7.38	
20	7.66	7.60	7.53	7.47	7.41	7.35	7.29	7.23	
21	7.50	7.44	7.38	7.32	7.26	7.20	7.14	7.23	
22	7.36	7.30	7.24	7.18	7.12	7.03	7.00	6.94	
23	7.22	7.16	7.10	7.05	6.99	6.93	6.87	6.81	
23 24	7.08	7.03	6.97	6.91	6.85	6.80	6.74	6.68	
25	6.95	6.90	6.84	6.79	6.73	6.67	6.62	6.56	
26	6.82	6.77	6.71	6.66	6.60	6.55	6.49	6.44	
27	6.70	6.65	6.59	6.54	6.49	6.43	6.38	6.32	
28	6.58	6.53	6.48	6.42	6.37	6.32	6.26	6.21	
29 30	6.47	6.42	6.36	6.31	6.26	6.21	6.15	6.10	
30	6.36	6.31	6.25	6.20	6.15	6.10	6.05	5.99	
31	6.25	6.19	6.14	6.09	6.04	5.99	5.94	5.89	
32	6.14	6.09	6.04	5.99	5.94	5.89	5.84	5.79	
33	6.04	5.99	5.94	5.89	5.84	5.79	5.74	5.69	
34	5.94	5.89	5.84	5.79	5.74	5.70	5.64	5.60	
35	5.84	5.79	5.74	5.69	5.64	5.59	5.55	5.50	
36	5.74	5.69	5.64	5.60	5.55	5.50	5.45	5.41	
37	5.64	5.60	5.55	5.50	5.46	5.41	5.36	5.31	
38	5.56	5.51	5.46	5.42	5.37	5.32	5.28	5.23	
39	5.46	5.41	5.37	5.32	5.28	5.23	5.18	5.14	
40	5.37	5.33	5.28	5.24	5.19	5.14	5.10	5.05	
41	5.28	5.24	5.19	5.15	5.10	5.06	5.01	4.97	
42	5.20	5.15	5.11	5.06	5.02	4.97	4.93	4.88	
43	5.12	5.07	5.03	4.98	4.94	4.90	4.85	4.81	
44	5.03	4.98	4.94	4.90	4.85 -/	4.81	4.77	4.72	
45	4.95	4.90	4.86	4.82	4.77	4.73	4.69	4.65	

#### 7.6.2 In Situ Measurements

The following steps are performed to take Hydrolab in situ measurements:

- 1. Remove the storage cup, install the circulator, and confirm that all connections are tight and that the circulator is operating freely (7.4.2).
- 2. Verify that the battery reading is above 11.5 volts.
- 3. Lower the sonde into the water, holding it horizontally to dislodge air bubbles that may be trapped in the specific conductance cell block.
- 4. Lower the sonde to the first depth of interest.
- 5. Switch the display to TEMP. Wait at least 5 minutes until readings are stable, indicating that the sonde has reached thermal equilibrium.
  - NOTE: The DO sensor is generally the slowest to reach thermal equilibrium. Carefully monitor DO stabilization and do not take measurements before equilibrium is reached.
- 6. Record temperature, specific conductance, pH, and DO.
- 7. Lower the sonde to the second depth of interest.
- 8. Wait at least 3 minutes for stabilization. Record temperature, specific conductance, pH, and DO.
- 9. Repeat steps 7 and 8 for all depths of interest.
  - NOTE: Never lower the instrument deeper than 1.5 m from the bottom, because contact could damage the sonde and disturb sediments.
- 10. Turn the unit off and raise the sonde to the surface. Remove the circulator and replace the storage cup. If possible, store the sonde unit with tap water in the storage cup. Lake water may be used, if necessary, until the unit is back at the base site.

# 7.7 Quality Assurance and Quality Control

# 7.7.1 Calibration Quality Control Check

A QCC for pH and specific conductance should be made following calibration and upon return from the field in the evening. The values are to be recorded without adjustment. This check should be performed in a temperature-controlled environment whenever possible.

Sulfuric acid solution is used as the QCC solution. Specific conductance and pH values are recorded and compared to the theoretical values for the solution (pH 4.03 at 25  $^{\circ}$ C, specific conductance 42  $\mu$ S/cm at 25  $^{\circ}$ C) as described below:

1. Rinse sensors three times with deionized water; discard each rinse.

- 2. Rinse sensors three times with the QCC solution. Discard each rinse.
- Fill calibration cup with QCC solution to a level just above the DO membrane. Record pH and specific conductance after allowing time for reading to stabilize (a minimum of 3 minutes).

NOTE: Make certain bubbles are not present in the bores of the specific conductance block. If bubbles are present, tap lightly to dislodge the bubbles, or pour out the QCC solution in the calibration cup and refill it.

- 4. Place a clean NBS-traceable thermometer in the calibration cup and stir the QCC solution gently. Allow time for equilibration and then compare the temperature reading from the thermometer to the reading given by the instrument. Record both values. Do not allow the thermometer to touch any surface while the temperature is being read.
- 5. If the observed value differs from the true value by more than  $\pm 0.20$  pH units or  $\pm 15~\mu$ S/cm for specific conductance, follow daily maintenance procedures (Section 7.8.1) and repeat calibration (Section 7.5). If results are still unsatisfactory, perform weekly maintenance procedures (Section 7.8.2), consult the troubleshooting directory (Section 7.8.3), and consult the Surveyor II manual. Calibrate and use a spare Hydrolab if the first Hydrolab cannot be calibrated for pH, specific conductance, or DO or if temperature values from the Hydrolab differ by more than 1 °C from an NBS-traceable thermometer.
- 6. Turn off Hydrolab, fill storage cup with tap water, and attach cup to sonde.

## 7.7.2 Field Quality Control Check

A QCC is performed at the field site before sampling operations begin, using the 0.0001 N  $H_2SO_4$  solution for pH and 147  $\mu S$ /cm KCl solution for specific conductance. Do not make any calibration adjustments in the field. Record data only. Do not store solutions or perform QCC in direct sunlight. Record the values on the field data form. The following steps must be completed:

- 1. At the lake, remove the storage cup; attach the calibration cup.
- 2. Rinse the sensors three times with deionized water.
- 3. Rinse the sensors three times with small portions of the 0.0001 N  $H_2SO_4$  QCC solution. Fill the calibration cup so that the solution level is over the DO membrane.
- 4. After stabilization, record the pH and conductance of the 0.0001 N  $H_2SO_4$  on the field data form. If the pH is ±0.20 pH unit from 4.00 or if the specific conductance is ±15  $\mu$ S/cm from 42, note this on the field data form.
- 5. Rinse the sensors three times with deionized water.
- 6. Rinse the sensors three times with small portions of the 147  $\mu$ S/cm KCl solution. Fill the calibration cup over the specific conductance sensor; be certain that no bubbles are trapped in the sensor bores.
- 7. After stabilization, record the pH and specific conductance of the KCl solution on the field data form. If conductance is  $\pm 15 \,\mu$ S/cm from 147, note this on the field data form.

8. Repeat rinse procedure with deionized water before immersing sensors in the lake.

### 7.7.3 Postsampling QCC

The postsampling QCC comprises the following steps:

- 1. Follow Section 7.7.1, steps 1 through 4, for the postsampling QCC for pH and specific conductance. Record the values on the calibration form.
- 2. Follow Section 7.5.4, steps 1 through 5, for the final check on DO measurement. Record the values on the calibration form, but make no adjustments with the toggle switches.

#### 7.8 Instrument Maintenance

### 7.8.1 Daily Maintenance

Hydrolab maintenance should be done after the postsampling QCC and before preparation for the next sampling day. Hydrolab maintenance includes the following steps:

- 1. Clean the instrument of dirt by rinsing several times with tap water. A warm detergent solution (Alconox) may be used if the sonde is extremely dirty. Fill the storage cup about half full with dilute detergent solution. Attach the storage cup and shake vigorously. It is very important that at least six rinses with tap water follow this treatment.
- 2. Inspect the sensor bores; remove any foreign matter with a cotton-tipped swab. Ensure that the threaded area and the rubber sealing ring of the sonde endcap are free of grit.
- 3. Rinse the sensors thoroughly with tap water.
- 4. Visually inspect for the following:
  - a. Wrinkles, perforations, or slackness in the DO membrane.
  - b. Bubbles in the electrolyte under the DO membrane.
  - c. Obstructions in the specific conductance cell block.
  - d. Coatings or precipitates on any sensor.

NOTE: Corrective actions for items a through d are provided in Section 7.8.2.

- e. Foreign matter in connectors and sockets, including cables (remove with cotton-tipped swab).
- f. Moisture in connectors and sockets (remove with CRC ether spray).
- 5. Service the pH reference electrode by wiping it with a piece of cotton moistened with alcohol or acetone. Fill black storage cap with 3 M KCl and install over the reference electrode tip.

- 6. Check the battery voltage; recharge if less than 11.5 volts. Always keep a spare charged battery for each Hydrolab.
- 7. Fill the storage cup with tap water and replace on sonde. Add Alconox to water to inhibit bacterial growth if the unit will be stored more than two weeks.
- 8. If daily maintenance procedures do not correct problems, follow the routine maintenance and troubleshooting protocols (Section 7.8.2 and Section 7.8.3, respectively).

### 7.8.2 Weekly Maintenance

Lightly lubricate the rubber internal mating surfaces of all sockets with silicone grease.

#### 7.8.2.1 Circulator Maintenance--

Weekly circulator maintenance consists of the following procedures:

- 1. Remove the impeller by lifting it from the bearing post.
- 2. Remove excess lubricant and grit from the cavity in the bottom of the impeller using a cotton-tipped swab.
- 3. Wipe off the bearing post and lightly relubricate it.
- 4. Turn the circulator on: verify smooth operation.

#### 7.8.2.2 Specific Conductance Cell Block Maintenance-

Remove oxidation from the specific conductance cell block as follows:

- 1. Protect the pH electrode by slipping a piece of flexible, thick-walled Tygon tubing over it.
- 2. Remove the cell block by removing the screw (use a small screwdriver). Remove the small O-rings on each of the six electrodes.
- 3. Examine the bores; remove any foreign matter with a cotton-tipped swab and warm detergent solution.
- 4. Burnish the electrodes with a strip of fine (400 grit) emery cloth. Polish the entire electrode, including the ends.

NOTE: Be careful not to scratch the pH electrode.

- 5. Wipe the electrodes with alcohol; flush out any residual grit with water.
- 6. Slip the O-rings back on the electrodes; push them down until they are flush against the sensor body. Replace any damaged O-rings.
- 7. Reposition the cell block on the sensor body; tighten the screw until the O-rings are compressed to approximately two-thirds of their uncompressed size.

8. Rinse the entire sonde well to remove clinging debris.

# 7.8.2.3 Dissolved Oxygen Sensor Maintenance--

If the DO membrane is slack, perforated, or torn, or if bubbles are evident under the membrane, the membrane should be replaced. This does not need to be done weekly, but the membrane should be checked daily. Replace as follows:

- 1. Remove the cylindrical membrane guard, the O-ring, and the membrane. Discard the membrane and shake out all electrolyte from the reservoir.
- 2. Hold the sonde at a 45-degree angle. Drip electrolyte slowly onto the lower surface of the reservoir so that it runs down the side wall and under the central electrode (anode). Be careful not to trap bubbles under the anode. When the anode is nearly covered, mount the sonde in a vise (sensors up), and fill until a large meniscus forms over the gold electrode (cathode).
- 3. Handle the new membrane by its edges with clean forceps or gloved hands. Hold one end of the membrane against the side of the DO sensor, (about 1 cm from the top) with your left thumb. Grasp the other end of the membrane between your right thumb and forefinger. In one smooth and rapid motion, stretch the membrane up and over the top of the sensor and secure the end with your left forefinger, keeping the membrane taut.
- 4. Check for air bubbles. Large bubbles indicate that capillary flow drained the meniscus away during stretching of the membrane (i.e., action was too slow). Return to the latter part of step 2 and repeat.
- 5. Roll the O-ring into place, securing the membrane. Check that the membrane is taut and free of wrinkles. Trim away excess membrane outside the O-ring with scissors. Replace the cylindrical membrane guard.
- 6. Allow at least 12 hours "aging" time prior to calibration and use.

### 7.8.3 Troubleshooting

The Surveyor II initiates a self-test procedure every time it is turned on. If a problem occurs in the self-test, the display will show an error message. Table 7-3 lists error messages and possible causes and remedies. The operator may perform several tests to help localize a problem. Problems and corrective actions are discussed in the Hydrolab Surveyor II manual. The self-test will not reveal calibration errors or individual sensor malfunctions.

#### 7.8.3.1 Sonde Response Test-

The sonde response test should be performed as follows:

- 1. Disconnect the data cable from the display unit and connect the I/O test cable in its place.
- 2. Set FUNCTION switch to OFF. Pull both ZERO and SLOPE switches simultaneously toward you. While holding the switches in that position, switch FUNCTION to TEMP.

Table 7-3. Troubleshooting Directory

	Symptom	Possible Cause and Remedy
a,	Error Messages	
	'IOrE'	DU not receiving databad DU ART board, bad SU ART board, bad cable, water in SUtest using I-O tester
	'IOSE'	DU not sending databad DU ART board.
	'H20'	Serious problem. Immediately flush transmitter 3 times with deionized water. Finish with 50/50 alcohol, deionized water, then air dry.
	Display flashing	Low battery (between 10.0 - 9.5 volts)recharge battery.
	??;oox	Parameter is out of rangecheck standard.
<b>)</b> .	Display trouble, odd or missing characters	Display test
<b>.</b>	Abnormal DO*, pH, specific conductance reading-temp, depth normal	Stray leakage to water. Wet battery, leaky connection, leaky cable
i.	Individual measurement problems	Prepare probes, check standard
).	Individual measurement problems, sensor failures	Call Hydrolab Service Department
•	Calibration difficulties	Prepare sensors, check standard
j.	Data logging troubles	Low battery, broken 12 IC cable, bad SU, bad 5100-A

<sup>\*</sup>DU = deck unit

- 3. If FAIL appears in the display, the malfunction is in the display unit. If PASS appears, proceed to the next step.
- 4. Reconnect the data cable to the display unit. Connect the I/O test cable to the submersible end of the data cable.
- 5. Repeat step 2. If FAIL appears in the display, the fault is in the data cable. If PASS appears, the problem is in the sonde.

NOTE: This test does not check the ground (pin C). This line must be checked for proper connection with a continuity tester.

# 7.8.3.2 Display Test--

This is a check of the liquid crystal display (LCD) only, to ensure that all segments are operating:

SU = sonde unit

DO = dissolved oxygen

- 1. Turn FUNCTION to OFF. Punch the ZERO and SLOPE toggle switches simultaneously away from you.
- 2. Switch FUNCTION to any sensor.
- 3. If the display is working, it will show -1.8.8.8. It will blink off, then on again, and selected sensor data will follow. If the display is not working or if it is incorrect, consult the manufacturer.

## 7.9 References

ASTM (American Society for Testing and Materials). 1984. Annual Book of ASTM Standards, Volume 11.01, Standard Specification for Reagent Water, D 1193-77 (reapproved 1983). ASTM, Philadelphia, Pennsylvania.

Hydrolab Corporation. 1984. Operations and Maintenance Manual for Hydrolab Surveyor II, Austin,TX.

# 8.0 Determination of pH (Lotic)

#### 8.1 Overview

The pH of an aquatic environment is regulated by abiotic (inorganic CO<sub>2</sub> equilibria, surficial geology, and anthropogenic pollutants) and biotic (photosynthesis, respiration, and decomposition) factors. A pH balance is usually maintained by the presence of buffering reactions within the aquatic system. A shift in this balance can cause chemical and biotic repercussions.

The pH is defined as the negative logarithm of the activity of hydrogen ions (H<sup>+</sup>). The H<sup>+</sup> activity is a measure of the "effective" concentration of hydrogen ions in solution; it is always equal to or less than the true concentration of hydrogen ions in solution. Values usually range from pH 1 to pH 14, with pH 1 most acidic, pH 7 neutral (at 25 °C), and pH 14 most alkaline. Each pH unit represents a tenfold change in H<sup>+</sup> activity (i.e., a pH 4 solution is 10 times as acidic as a pH 5 solution).

When the pH of a sample solution is measured, the hydrogen ions come into equilibrium with the ion exchange surface (glass) of a calibrated pH electrode, which creates an electrical potential. This voltage difference is measured by the pH meter in millivolts, then is converted and displayed as pH units.

# 8.1.1 Scope and Application

This method is applicable to the determination of pH in samples from flowing waters of low ionic strength. This procedure is similar to the one utilized in NSS. It assumes that pH measurements will be made at streamside (Section 6.0).

Measurement of stream water pH may be done in situ. However, streaming potential effects may reduce the accuracy of pH measurements from waters in motion. For this reason, measurements are generally obtained from either closed chambers (U.S. EPA, 1987) or open beakers, with portable pH meters. The AERP surveys relied on Beckman model 121 meters and Orion-Ross model BNC-8104 glass electrodes to measure the pH of stream waters. The method described here assumes that the Beckman meter and Ross electrode are used. This method, however, can be used with modification for other portable instrumentation meeting equivalent specifications. This method is applicable to systems other than lotic systems.

The applicable pH range is 3.0 to 8.0 units. The range may be extended with use of a wider range of pH calibration standards and pH QCC solutions.

# 8.1.2 Summary of Method

The pH meter and electrode are calibrated, and the quality of measurements determined, prior to base site departure. At streamside, the meter is checked against a pH 4.00 QCC solution and a pH 7.00 buffer standard. If the meter does not fall within specified limits for each check, it should be recalibrated. The water sample used to determine pH is pumped from the stream

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through Tygon tubing into a beaker. A glass electrode and an automatic thermocompensator (ATC) probe are placed into the sample, and its pH is displayed on the meter's digital readout.

#### 8.1.3 Interferences

No interferences are known within the range commonly encountered in low ionic strength waters.

### 8.1.4 Safety

The calibration standards, sample types, and reagents used in this method pose no hazard to the sampler. General safety guidelines for samplers operating in flowing waters and under remote conditions are provided in Section 6.5.

# 8.2 Sample Collection, Preservation, and Storage

Water samples for pH determinations are collected with a peristaltic pump and food-grade Tygon tubing. The tubing is attached to the end of a fiberglass extension pole and placed in the stream at midchannel and middepth. After the tubing is purged for one to two minutes, a 250-mL sample beaker is rinsed three times with stream water. A sample of 150 to 200 mL is then pumped into the beaker. This sample and a second, collected in the same manner, are used to determine stream pH.

# 8.3 Equipment and Supplies

## 8.3.1 Equipment

Beckman 121 portable pH meter or equivalent.

## 8.3.2 Apparatus

- 1. Meter operation manual.
- 2. ATC probe.
- 3. Orion-Ross model BNC-8104 combination glass electrode.
- 4. Wash bottle (1 L).
- Six 250-mL bottles for field rinse, pH 4.00 QCC solutions, and pH 4.00 and 7.00 buffer standards.
- 6. NBS-traceable thermometer.
- 7. Watch or stopwatch.

## 8.3.3 Reagents and Consumable Materials

- 1. Calibration and field data forms.
- 2. Deionized water--used in all preparations should conform to ASTM specifications for Type I reagent grade water (ASTM, 1984).
- 3. pH calibration standards--commercially available pH buffers (NBS-traceable values of pH 4.00 and pH 7.00 at 25 °C).
- 4. pH 4.00 QCC solution; 0.0001 N--1 mL of 0.1 N H<sub>2</sub>SO<sub>4</sub> diluted to 1 L with deionized water. Prepare daily.
- 5. Six 250-mL disposable beakers.
- 6. Electrode filling solution (3 M KCI).

## 8.4 Preparation

- NOTE: It is recommended that all personnel operating pH meters be familiar with the operating procedures prior to using these meters. The pH meter *must* remain dry. The pH meter should be enclosed in plastic with desiccant packets and should be checked daily for moisture problems.
- NOTE: The Orion-Ross pH electrode has a glass bulb. Care should be taken in handling the electrode to prevent shock to the bulb. The electrode should always be carried upright in a padded case or vest pocket.
- 1. Lower the pH electrode's fill hole collar to uncover the opening.
- 2. Make sure the pH electrode is properly conditioned for use. Refer to electrode instruction manual. If response time is reduced to unacceptable levels, recondition the electrode as described in Section 8.8.
- 3. Make sure the electrode and ATC probe are properly connected.
- 4. Verify that the reference filling solution is at least 3 cm above the sample line. If not, adjust electrode and add filling solution as required.
- 5. Observe battery and probe error signal locations for indication of problems. If either appear, troubleshoot as needed (see manufacturer's manual).

### 8.5 Calibration and Standardization

NOTE: Refer to Figure 8-1, Flowchart for pH Meter Calibration.

Using NBS-traceable buffers, meters should be recalibrated each morning to pH 4.00 and pH 7.00. Calibration should be checked, first by reading the pH 4.00 and pH 7.00 buffers, then by reading the lower ionic strength pH 4.00 QCC solution.

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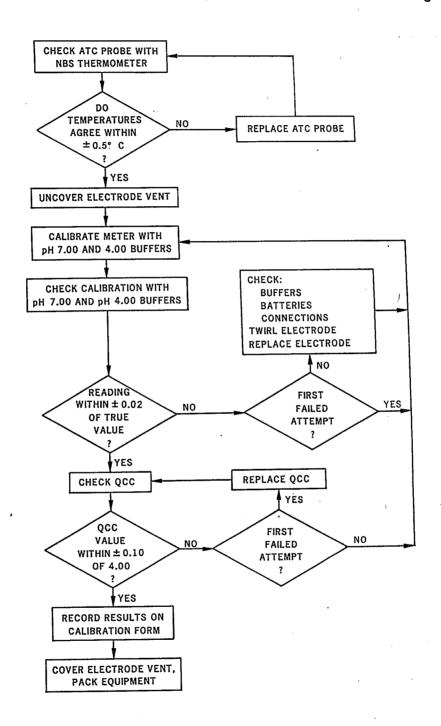


Figure 8-1. Flowchart for pH meter calibration.

Time for equilibration of the pH electrode varies depending on the ionic strength of the solution being measured. The criterion for a stable reading is no change of more than 0.01 pH unit during a 60-second period. All measurements of pH during calibration, calibration checks, QC checks, and sample measurement use this criterion for a stable reading.

The pH meter switches off automatically after a period during which no buttons are pressed. If the display switches off, press the "pH" key to restart the display.

#### 8.5.1 ATC Probe Check

- 1. Immerse the electrode, ATC probe, and NBS-traceable thermometer into the rinse beaker of pH 7.00 buffer.
- 2. Press the "pH" key and read the temperature on the display. The two temperature readings should agree to within 0.5 °C.
- 3. If they do not agree, stir the solution and check the temperature again after a short pause for equilibration.
- 4. If they still do not agree, either the ATC probe or the meter is malfunctioning. Replace the ATC sensor and check the temperature again.
- 5. If this does not result in agreement with the NBS-traceable thermometer, refer to the instrument manual for troubleshooting guidance.

# 8.5.2 Calibration with NBS-Traceable Buffers

- NOTE: The "Auto-lock" key on the Beckman pH meter is used only during standardization, and then only after a stable reading is attained. All measurements of samples, calibration checks, and QC checks *must* be made with "Auto-lock" OFF.
- NOTE: In the field, buffer solutions are carried in two 125-mL bottles. To avoid cross-contamination, clearly mark "rinse" on one bottle and its cap. Mark "test" on the other bottle. Use 250-mL plastic beakers for initial standardization at the base site. These beakers should also be clearly marked "rinse" and "test."
- 1. Press the "Clear" key on the pH meter.
- 2. Rinse the electrode and probe with deionized water and immerse the electrode and ATC probe in the "rinse" beaker of pH 7.00 buffer.
- 3. Gently swirl the electrode and ATC probe in the buffer for 30 seconds.
- 4. Move the electrode and ATC probe to the "test" beaker of the same buffer. Do not swirl.
- 5. Press the "Standard" key.
- 6. Turn the "Auto-lock" feature OFF.

- 7. Observe the pH display. When the display is stable (as defined in the second paragraph of Section 8.5) for 60 seconds, turn "Auto-lock" ON. When the reading locks, the standard has been stored in memory.
- 8. Rinse the electrode and ATC probe in deionized water.
- 9. Place the electrode and ATC probe in the rinse beaker of pH 4.00 buffer.
- 10. Repeat steps 3-8 above.

#### 8.6 Procedure

NOTE: Refer to Figure 8-2, Flowchart for field pH measurement.

# 8.6.1 Field Quality Control Check

NOTE: Leave the "Auto-lock" OFF during this entire procedure.

- Check the connection of the electrode and ATC probe. Twirl the electrode gently to remove bubbles. Remove the KCI-filled storage cap and lower the collar on the reference solution fill hole.
- 2. Rinse the electrode with deionized water.
- . 3. Conduct an initial QC check by using pH 4.00 QCC solution. If the QC check is unacceptable, recalibrate, then recheck the QC. If it is still unacceptable, calibrate and use the spare electrode. If the spare electrode will not meet standards, the data must be qualified.
  - 4. Rinse the electrode and ATC probe with deionized water.
- 5. Record QCC pH and temperature readings on the field logbook form and record the pH reading on the field data form.

# 8.6.2 Sample Measurement

- 1. Perform initial field quality control check (see Section 8.6.1).
- 2. After purging the tubing and rinsing the beaker three times, pump a 150- to 200-mL sample of stream water into a 250-mL beaker.
- 3. Press the "pH" key and swirl the electrode and ATC probe in the sample for 3 minutes.
- 4. Collect a fresh sample and transfer the electrode and ATC probe. Let it sit, unswirled, for 2 minutes, then begin watching for a stable reading (±0.01 pH units for one minute). Record the pH, temperature, and time required for stabilization on the field data form.
- 5. Remove the electrode and ATC probe in such a way as to prevent them from touching the ground or other surfaces and discard the sample. Collect a fresh 150-mL sample.

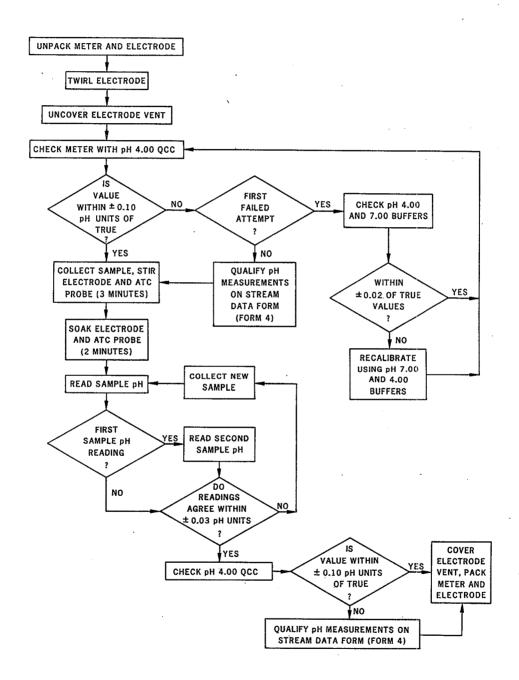


Figure 8-2. Flowchart for field pH measurement.

- 6. Immerse the electrode and ATC probe in a fresh sample. When the reading is stable, record the pH, temperature, and time required for stabilization on the field logbook form.
- 7. Repeat steps 4 and 5 until two successive pH readings agree within 0.03 pH units. The first reading is not used for successive comparisons, thus there are at least 3 sample readings and stabilization times on the field data form. Record final pH and temperature readings on the field data form.
- 8. If a duplicate sample is to be taken at this stream site, repeat steps 1-6. Record duplicate pH and temperature readings in the appropriate spaces on the stream data form.
- 9. Perform a final QCC (Section 8.6.3) at each sampling site.

### 8.6.3 Post-Deployment Quality Control Check

- 1. Conduct a final pH 4.00 QCC check after the final sample pH determination at each site (Section 8.6.1). Record pH and temperature readings on the field data form.
- 2. Press the "OFF" button on meter. Do not press the "Clear" key.
- 3. Replace the cap filled with 3 M KCI on the tip of the pH electrode and raise the collar on the pH reference solution fill hole.

# 8.7 Quality Assurance and Quality Control

Quality assurance and quality control procedures related to measuring pH are described in the following subsections.

#### 8.7.1 Calibration Check

A calibration check should be conducted immediately following calibration to verify the accuracy of the calibration values stored in memory.

NOTE: Turn "Auto-lock" OFF during this entire procedure.

- 1. Swirl the electrode and ATC probe in the "rinse" beaker of pH 7.00 buffer for 30 seconds.
- 2. Move to the "test" beaker. Do not swirl.
- 3. Press the "pH" key on the meter and observe the pH display. When the reading is stable for 60 seconds, record the pH and temperature on the calibration form.

NOTE: If recalibrating in the field, record the pH and temperature on the field data form.

- 4. If the displayed reading differs from the theoretical value at the measured temperature (Table 7-1, Section 7.0) by greater than 0.02 pH units, recalibrate using both buffers.
- 5. Rinse the electrode and ATC probe in deionized water and proceed.
- 6. Repeat steps 1-5 for the pH 4.00 buffer.

## 8.7.2 pH Quality Control Check

NOTE: This procedure is conducted immediately following a successful initial calibration check to ensure the accuracy of the calibration for relatively low ionic strength, unbuffered solutions.

NOTE: QC solution (Section 8.3.3) has a theoretical pH of 4.00 at 25 °C.

NOTE: Beakers marked "QCC solution rinse" and "QCC solution standard" are used for morning calibrations at the base site.

NOTE: Leave the "Auto-lock" OFF during this entire procedure.

- 1. Swirl the electrode and ATC probe in the "QCC solution rinse" beaker for 3 minutes.
- 2. Move to the "QCC solution test" beaker. Do not swirl.
- 3. Press the "pH" key on the meter.
- 4. Observe the pH display. Wait two minutes, then begin timing the stability of the reading. When the reading is stable for 60 seconds (±0.01 pH units), record pH and temperature on the calibration form.
- 5. If the displayed reading differs from 4.00 by greater than 0.10 pH units, rinse the probe and check again with a fresh beaker of QCC solution.
- 6. If the value is still unacceptable, prepare a fresh QCC solution and begin again.
- 7. If the value is still unacceptable, clear and recalibrate using both buffers.
- 8. When an acceptable QCC solution value has been obtained, push the "OFF" key on the meter and pack for transport to streamside.

NOTE: Calibration data are retained by the meter. Do not press the "Clear" key, or calibration data will be erased. To restart pH measurements when the instrument is off, press the "pH" key.

# 8.8 Routine Maintenance and Care

- The meter should be sealed in a plastic bag containing a desiccant package (e.g., silica gel). The bag should be placed in a second sealable plastic bag for transport.
- 2. The meter should be stored so as to minimize physical shock during transport.
- 3. Avoid exposing the meter and electrodes to extremes of temperature or to direct sunlight.
- 4. Electrodes should be kept upright as much as possible, especially during transport.
- 5. The electrode and ATC probe should be carried wrapped in a plastic bag.
- 6. Keep the electrode filled with 3 M KCI.

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- 7. Always carry a spare electrode which is known to be fully functional.
- Approximately once a week, or more often if electrode response is sluggish or if the meter will not standardize using a specific electrode, the following procedure is recommended:
  - Carefully drain the filling solution from the outer chamber of the electrode through the vent using a syringe equipped with a small diameter tube.
  - b. Using the syringe, the small diameter tubing, and deionized water, rinse and then drain the chamber thoroughly.
  - c. Rinse the chamber by filling, agitating, and draining it with 3 M KCI, then fill the outer chamber with fresh 3 M KCI through the vent.
- 9. If the electrode response does not improve after completion of step 8, electrode etching is recommended. Electrodes should be returned to the central processing laboratory for etching. If this is not possible, they may be etched in the field by using the following process:
  - a. Drain the filling solution from the electrodes.
  - b. Rinse the filling chambers with deionized water and drain.
  - c. Refill with deionized water.
  - d. Prepare a 50 percent (W/V) NaOH solution by slowly adding 30 g NaOH to 30 mL deionized water.
  - e. Stir the solution with the electrodes to dissolve the NaOH.
  - f. Stir the solution another 2 minutes with the electrodes.
  - g. Rinse the electrodes with deionized water.
  - h. Rinse the electrodes in pH 7.00 buffer for 2 minutes.
  - i. Drain the deionized water form the filling chamber.
  - j. Refill with 3 M KCl, agitate the electrodes and drain.
  - k. Refill with 3 M KCl, and twirl the electrodes overhead by their leaders to remove bubbles.
  - NOTE: Etch electrodes in groups of three. Prepare a fresh NaOH solution for each group of electrodes.
  - CAUTION: NaOH is extremely caustic. The solution is exothermic, and the solution will become very hot. Prepare the etching solution in a very well ventilated room, avoid breathing the fumes, and exercise extreme caution.

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# 8.9 References

- ASTM (American Society for Testing and Materials). 1984. Annual Book of ASTM Standards, Volume 11.01, Standard Specification for Reagent Water, D 1193-77 (reapproved 1983). ASTM, Philadelphia, Pennsylvania.
- U.S. EPA (Environmental Protection Agency). 1987. Handbook of Methods for Acid Deposition Studies: Laboratory Analyses for Surface Water Chemistry. EPA 600/4-87/026. U.S. EPA, Office of Research and Development, Washington, D.C. 342 pp.

# 9.0 Determination of Specific Conductance (Lotic)

#### 9.1 Overview

Specific conductance or conductivity is a measure which often can be linearly correlated with the ionic strength of a solution. Specific conductance also can be used to generate a synthetic ionic balance of a solution. This balance can be used as a check of measured cation and anion concentrations.

## 9.1.1 Scope and Application

This method, which is similar to the one utilized in NSS, is applicable to the determination of specific conductance in samples from waters of low ionic strength.

Streaming potential is generally not a problem during conductivity measurements. For this reason, stream water measurements usually are taken in situ, but also may be obtained from open beakers. Portable conductivity meters are used most commonly. The AERP surveys relied on Yellow Springs Instruments Co. (YSI) model 33 S-C-T meters and model 3310 conductivity/ temperature probes to measure specific conductance. The method described here assumes that the YSI meter is used and stream waters are sampled in situ. The method can be modified and used for other instrumentation meeting equivalent specifications. The method is not limited to lotic systems. Although inefficient under many circumstances, in situ specific conductance of a lake water sample may be determined with some modification of this procedure.

The applicable range of measurements taken with the YSI is 2.5 to 50,000  $\mu$ S/cm ( $\mu$ mhos/cm) at 25 °C (YSI, 1983). The specific conductance of most AERP-sampled streams ranged between 10 and 500  $\mu$ S/cm (at 25 °C) (Kaufmann et al., 1988). However, only measurements in the range of 50 to 1000  $\mu$ S/cm (at 25 °C) are quality checked for accuracy with this method. Because the YSI meter calibration is preset, the range cannot be extended. However, the range that is quality assured can be extended by measurement verification with a wider range of QCC solutions.

## 9.1.2 Summary of Method

The YSI meter and probe are calibrated at the factory and refined adjustments are not possible at the base station. The quality of specific conductance and temperature measurements is determined with low-range, mid-range, and high-range QCC solutions prior to sampling. There is no true meter calibration for this method. At streamside, the meter is checked against a single, low-range QCC solution. A probe is placed into the stream and conductance values are displayed on the meter's analog readout. Conductance readings are not temperature compensated. All values are adjusted (based on sample temperature and a correction table) to specific conductance at a reference temperature of 25 °C.

### 9.1.3 Interferences

Temperature variations represent the major source of potential error in specific conductance determinations. To minimize this error, meter quality control checks are conducted at the base site under controlled temperatures, readings are adjusted relative to 25 °C, and temperature measurements are checked against an NBS-traceable thermometer.

Natural surface waters contain substances (oils, humic and fulvic acids, suspended solids) that may build up on metal surfaces of the conductivity probe. Such a build up interferes with the operation of the electrode and should be removed periodically, following the manufacturer's instructions (YSI, 1983).

Measurements with the conductivity/temperature probe can be affected by objects in close proximity. The manufacturer recommends that metal and nonmetal materials (including the stream bottom) be kept at least 6 and 2 inches, respectively, away from the probe during all readings.

### 9.1.4 Safety

The calibration standards, sample types, and reagents used in this method pose no hazard to the sampler. General safety guidelines for samplers operating in flowing waters and under remote conditions are provided in Section 6.5.

# 9.2 Sample Collection, Preservation, and Storage

Since stream water specific conductance generally is determined in situ, sample collection, preservation, and storage are not applicable. Specific conductance is determined from an electrode suspended at midchannel and middepth. Water samples may be collected from streams and lakes with a peristaltic pump and Tygon tubing. A Van Dorn sampler also may be used for collection of lake water samples from specified depths. A water volume of at least 200 mL is required to measure specific conductance in a beaker. After the meter is prepared, the probe is placed into the sample, and electronic and thermal equilibria are established. Measurements are then taken.

# 9.3 Equipment and Supplies

# 9.3.1 Equipment

- 1. YSI model 33 S-C-T portable meter, or equivalent.
- 2. YSI model 33l0 probe with cable, or equivalent.

# 9.3.2 Apparatus

- Meter operation manual.
- NBS-traceable thermometer.
- 3. Two 250-mL bottles for field rinse and test QCC (74  $\mu$ S/cm) solutions.

4. Wash bottle (1 L).

#### 9.3.3 Consumable Materials

- 1. Calibration and field data forms or logbook.
- 2. Six 250-mL disposable beakers for rinse and test QCC (74, l47, and 7l8 µS/cm) solutions.

#### 9.3.4 Reagents

- 1. Deionized water--used in all preparations; should conform to ASTM specifications for Type 1 reagent grade water (ASTM, 1984).
- 2. 74  $\mu$ S/cm field specific conductance QCC solution (0.0005 N KCl)--0.5 mL of 1 N KCl diluted to 1 L with deionized water.
- 3. 147 μS/cm specific conductance calibration check solution (0.001 N KCl)--1 mL of I N KCl diluted to 1 L with deionized water.
- 718 µS/cm specific conductance calibration check solution (0.005 N KCl)--5 mL of 1 N KCl diluted to 1 L with deionized water.

## 9.4 Preparation

The following procedure should be performed daily. If the instrument is subjected to physical shock, repeat this preparation process. Note that the probe should always be stored in deionized water between uses.

- 1. Check probes for outward signs of fouling. Do not touch the electrodes inside the probe with any object.
- 2. Plug the probe securely into the instrument jack.
- 3. Adjust the meter to "ZERO" with the set screw on the meter face so the needle coincides with "0" on the scale.
- 4. Turn the mode control to "REDLINE." Adjust the redline control so that the needle will line up with the red line on the meter scale. If alignment cannot be achieved, replace the batteries.

### 9.5 Calibration and Standardization

NOTE: Refer to Figure 9-1, Flowchart for conductivity meter calibration.

Because the YSI conductivity meter is calibrated at the factory, temperature and specific conductance measurements can only be compared to known standards to determine accuracy. This is really a QCC and is termed meter calibration check. It should not be confused with true calibration procedures. Meter zero and redline adjustments are made, but they do not relate to any standard or known solution values.

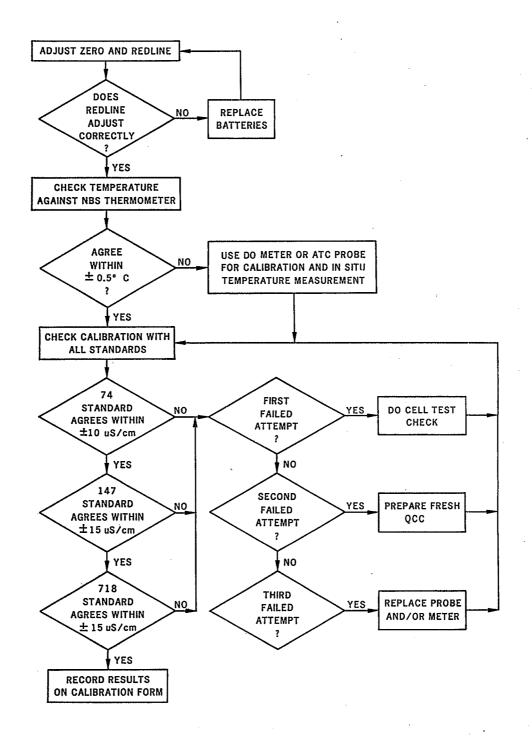


Figure 9-1. Flowchart for conductivity meter calibration.

Perform initial temperature and calibration checks daily, prior to departure for the field sites. All specific conductance readings should be recorded on calibration and field data forms. This procedureutilizes and records both temperature-uncompensated and temperature-compensated specific conductance values, where applicable. This practice reduces the probability of calculation errors resulting in unrecoverable losses of data. Temperature correction factors (at 25 °C) used for calculation of theoretical specific conductance values are listed in Table 9-1.

Table 9-1. Factors for Converting Specific Conductance of Water to Values at 25 °C (Based on 0.1 N KCl and 0.01 N NaN03 Solutions)<sup>a</sup>

.c	Factor	•c	Factor	*c	Factor	•c	Factor	•c	Facto
32	0.89	26	0.98	20	1.10	14	1.24	8	1.42
31	0.90	25	1.00	19	1.12	13	1.27	7	1.46
30	0.92	24	1.02	18	1.14	12	1.30	6	1.50
29	0.93	23	1.04	17	1.16	11	1.33	5	1.54
28	0.95	22	1.06	16	1.19	10	1.36	4	1.58
27	0.97	21	1.08	15	1.21	9	1.39	3	1.62

Wetzel, R. G., and G. E. Likens, 1979. Limnological Analyses. W. B. Saunders Co., Philadelphia.

When making measurements, the entire electrode and thermistor (located on top of the electrode) should be fully submerged, but the electrode should not rest on the bottom of the beaker or stream channel.

#### 9.5.1 Temperature Check

- 1. Rinse the probe and NBS-traceable thermometer with deionized water before and after submersion in QCC solutions.
- 2. Immerse the probe in the rinse beaker of the 74  $\mu$ S/cm standard. Be certain that the thermistor is fully submerged.
- 3. Set the mode switch to "TEMP."
- Compare the meter reading with that obtained by using an NBS-traceable thermometer. The readings should agree within 0.5 °C of each other. Record values on the calibration form.
- 5. If temperature readings do not agree, do not use the conductivity probe to measure QCC solution temperature or in situ temperature at the sample site. Use an alternate method for temperature measurements. Make note of this change on the field data form.

#### 9.5.2 Initial Calibration Check

The base site calibration check consists of comparing specific conductance values of the three QCC solutions to theoretical values that have been corrected for temperature. If measurements do not fall within prescribed limits for each QCC solution, compare the meter values to another meter and probe. If readings on the other meter are acceptable, use the other meter or troubleshoot the original meter. If readings are unacceptable with the second meter, replace

check solutions and check again. If readings for the two meters are still unacceptable, troubleshoot one or both as described in Section 9.8.

- 1. Rinse the probe with deionized water.
- 2. Immerse the probe in the rinse beaker containing 74  $\mu$ S/cm QCC solution. Agitate the probe slightly. Remove the probe from the rinse beaker.
- 3. Immerse the probe in the standard beaker containing fresh solution. Determine the temperature and record it on the calibration form. Set the mode selector to the "X1" scale.
- 4. Read the conductivity and record it as uncompensated conductivity on the calibration form. Calculate the temperature-compensated specific conductance and record it on the calibration form. The temperature compensated value should be 74  $\mu$ S/cm  $\pm$  7  $\mu$ S/cm.
- 5. Repeat steps 1-4, using the 147  $\mu$ S/cm specific conductance standard solution. The temperature corrected value should be 147  $\mu$ S/cm ± 15  $\mu$ S/cm.
- 6. Repeat steps 1-4, using the 718  $\mu$ S/cm specific conductance standard solution. The temperature corrected value should be 718  $\mu$ S/cm ± 72  $\mu$ S/cm.

#### 9.6 Procedure

The probe should be fully submerged in the stream water and should not contact the stream bottom. Note that in coastal areas, some streams may be affected by tidal influences. During NSS, when corrected specific conductance at a site was determined to be greater than 500  $\mu$ S/cm in inland streams, sampling was discontinued. If corrected specific conductance was determined to be greater than 250  $\mu$ S/cm in coastal streams, sampling also was discontinued. Sampling sites were moved upstream to the point where specific conductance first fell below 250  $\mu$ S/cm.

- 1. Perform initial field quality control check (see Section 9.7.1).
- 2. Immerse the probe in the flowing portion of the stream, downstream from the pump intake tubing.
  - NOTE: The probe should be fully immersed in the stream flow but should not be touching bottom. This can be accomplished by placing it across the sampling boom.
- 3. Set "MODE" selector to "X1" scale and read specific conductance. Record the value on the field data form. Calculate the temperature compensated value or, alternately, the conversion may be done by computer at a later date.
- 4. Between sampling sites, perform an additional field quality control check.

## 9.7 Quality Assurance and Quality Control

## 9.7.1 Field Quality Control Check

Perform the QCC at each sampling site before and after in situ specific conductance determinations. Record uncompensated and compensated QCC solution measurements on the field data form.

NOTE: Refer to Figure 9-2, Flowchart for field specific conductance measurement.

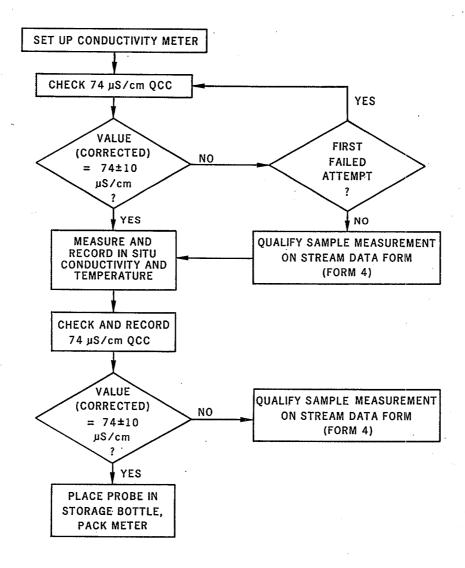


Figure 9-2. Flowchart for field specific conductance measurement.

- 1. Follow the procedure in Section 9.5.2, steps 1-4 for Initial Calibration Check, using the 74  $\mu$ S/cm QCC solution carried into the field in two 250-mL plastic bottles, one labeled "rinse" and one labeled "test." Record uncompensated QCC solution conductivity and temperature values in the field logbook and on the field data form.
- Calculate the compensated QCC solution specific conductance and record on the field data form.
  - a. Compensated QCC solution reading must be 74  $\pm$  10  $\mu$ S/cm. If not, repeat QCC solution check.
  - b. If an acceptable value is not obtained for the second QCC solution, proceed with the in situ determination and final QCC.
  - c. If either the initial *or* the final QCC solution measurement does not fall within acceptable limits, qualify all specific conductance values on the stream data form associated with the unacceptable QCC solution value(s).

### 9.7.2 Post-Deployment Quality Control Check

After returning from the field, if any QCC solution measurements made during site sampling operations were outside specified limits, repeat the calibration and QCC solution procedures (Section 9.5), perform maintenance, or troubleshoot the meter according to Section 9.8, and the manufacturer's operation manual.

### 9.8 Instrument Maintenance

#### 9.8.1 Routine Maintenance

Refer to the instrument manual for probe cleaning instructions.

- 1. Store the probe in deionized water.
- 2. Before using a probe which has been stored dry, soak the probe in deionized water for 24 hours.
- 3. Always turn off the meter after use.
- 4. Keep the meter dry.

#### 9.8.2 Troubleshooting

If acceptable QCC solution values are not obtained, compare values read by the other sampling teams. If the readings obtained by other teams are also unacceptable, replace the QCC solution in question. If the other teams are obtaining acceptable QCC readings, troubleshoot the meter and probe.

- 1. Rinse the probe well and recheck the questionable solution.
- 2. Recheck "REDLINE" and replace the batteries, if necessary.

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- 3. Using the 718  $\mu$ S/cm with the meter set to the "X10" scale, press the "CELL TEST" button. If the reading falls more than two percent, the probe is fouled. Clean the probe according to the manufacturer's instructions.
- 4. If these measures do not remedy the problem, replace the meter or probe, or both. To determine whether it is the meter or the probe that is malfunctioning, switch probes with another meter for which acceptable QCC readings have been obtained. If values obtained are still unacceptable, the meter is malfunctioning. If acceptable readings are obtained, the original probe is malfunctioning.
- 5. If the meter will not meet QCC for the 147 or 718  $\mu$ S/cm standards and if no replacement meter/probe is available, all field data forms for the day should include this information.

### 9.9 References

- ASTM (American Society for Testing and Materials). 1984. Annual Book of ASTM Standards, Volume 11.01, Standard Specification for Reagent Water, D 1193-77 (reapproved 1983). ASTM, Philadelphia, Pennsylvania.
- Kaufmann, P., A. Herlihy, J. Elwood, M. Mitch, S. Overton, M. Sale, J. Messer, K. Reckhow, K. Cougan, D. Peck, J. Coe, A. Kinney, S. Christie, D. Brown, C. Hagley, and Y. Jager. 1988. Chemical Characteristics of Streams in the Mid-Atlantic and Southeastern United States. Volume I: Population Descriptions and Physico-Chemical Relationships. EPA 600/3-88/021a. U.S. Environmental Protection Agency, Washington, D.C.
- Yellow Springs Instrument Company. 1983. Instructions for YSI Model 33 and 33M S-C-T Meters. Yellow Springs Instrument Company, Inc., Yellow Springs, Ohio.

# 10.0 Determination of Dissolved Oxygen (Lotic)

### 10.1 Overview

Dissolved oxygen (DO) is a measure of the amount of oxygen concentration dissolved in solution. In natural waters, minimal concentrations of oxygen are essential for survival of most aquatic organisms. Measures of DO and temperature are used to assess water quality and the potential for healthy aerobic organism populations.

### 10.1.1 Scope and Application

This method is applicable to the determination of DO in natural waters. The procedure is similar to the one utilized in the NSS.

Moving waters are generally considered essential for accurate DO determinations. For this reason, stream water measurements are usually taken in situ. Measurements of DO from beakers, biological oxygen demand (BOD) bottles, or lake strata can be accomplished with various probe attachments and/or manual stirring procedures. Portable DO meters are commonly utilized in stream studies. The AERP surveys relied on Yellow Springs Instrument Co. (YSI) model 54A oxygen meters and model 5739 dissolved oxygen and temperature probes to measure DO. The method described here assumes that the YSI meter is used and steam waters are sampled in situ. The method can also be used with modification for other instrumentation meeting equivalent specifications. The method is not limited to lotic systems. In situ DO measurements in lake waters can be taken with minor changes in the procedure.

The applicable range of measurements taken with the YSI DO meter is 9 to 20 mg/L (ppm  $O_2$ ) (YSI, 1980). Because calibration is relatively simple, the meter can be calibrated either at the base station or at the field site. Calibration accuracy is verified with a QCC at the base station.

## 10.1.2 Summary of Method

The DO meter and probe are air calibrated at the base station prior to field activities, on site, and upon return. After each base station calibration, the quality of DO and temperature measurements is determined by comparing readings to theoretical concentrations of air-saturated deionized water. At streamside, the meter is recalibrated at ambient temperatures. The probe is placed into the stream and the DO values are displayed on the meter analog readout. Dissolved oxygen readings are adjusted to compensate for temperature and pressure (depth).

#### 10.1.3 Interferences

Sources of potential error in DO determinations include low battery voltage, changing instrument position after on-site calibration, lack of water flow across the membrane, loss of probe membrane integrity (bacterial colonization or punctures), improper calibration, storage of probe in deionized water, and poor membrane replacement techniques. Proper measurement and maintenance procedures should alleviate these problems.

Natural surface waters contain gases which may contaminate and tarnish the gold cathode, causing erroneous measurements. If the probe is used for long periods with a loose fitting membrane, silver may plate the cathode. These buildups can interfere with the operation of the sensor and should be removed according to the manufacturer's instructions (YSI, 1980).

### 10.1.4 Safety

The calibration standards, sample types, and reagents used in this method pose no hazard to the sampler. General safety guidelines for samplers operating in flowing waters and under remote conditions are provided in Section 6.5.

## 10.2 Sample Collection, Preservation, and Storage

Stream water DO is generally determined in situ, hence procedures for sample collection, preservation, and storage are not applicable. Dissolved oxygen is measured from a probe suspended at midchannel and middepth. The only requirement for stable readings is sufficient water flow (or probe stirring) to continuously replace oxygen at the water/membrane interface. After the probe is placed into the sample and electronic and thermal equilibrium is established, measurements are taken.

## 10.3 Equipment and Supplies

Sections 10.3.1 through 10.3.3 list the equipment, apparatus, and other materials used in the procedure described here.

### 10.3.1 Equipment

- 1. YSI model 54A portable meter, or equivalent.
- 2. YSI model 4739 probe with cable, or equivalent.

#### 10.3.2 Apparatus

- 1. Meter operation manual.
- 2. NBS-traceable thermometer.
- 3. Probe calibration chamber.
- 4. 1-gallon plastic bottle.
- 5. 3-gallon bucket.
- 6. Aquarium pump, air stone, and air tubing.
- 7. Calculator.
- 8. Watch.

## 10.3.3 Reagents and Consumable Materials

- 1. Calibration and field data forms.
- 2. Replacement membranes.
- 3. Probe electrolyte.
- 4. Tap water.

## 10.4 Preparation

The DO meter is prepared daily, prior to calibration and on-site measurements. The probe is always stored in tap water between uses. Stream water may be used, but should be replaced with tap water when available. If the instrument is subjected to physical shock, repeat the following preparation process:

- 1. Adjust the zero, using the screw on the meter face with the selector switch in "OFF" position.
- 2. Turn the selector to "REDLINE." Adjust the "REDLINE" knob to align the needle with the red line on the meter panel.
- 3. Turn the selector to "ZERO." Adjust the needle to the "0" value with the "ZERO" control knob.
- 4. Check the membrane on the probe for air bubbles.

### 10.5 Calibration and Standardization

Zero and redline adjustments to the meter are made, as noted above, prior to calibration and field measurements. Perform calibration daily, prior to departure for the field sites, prior to sample measurement, and again upon return to the base station. All DO readings and calculated intermediate values should be recorded on calibration and field data forms. Calibration procedures utilize temperature and altitude to calculate theoretical DO in water-saturated air.

NOTE: Refer to Figure 10-1 (Flowchart for DO meter calibration).

#### 10.5.1 Calibration

- 1. Attach the moist air calibration chamber to the probe, release end clamp, and immerse the probe in a water bath (bucket or stream). Turn the meter to "REDLINE." Equilibrate the probe for 15 minutes.
- 2. Turn the selector switch to "TEMP," read the temperature of the chamber, and determine the saturation value from the O<sub>2</sub> solubility table (Table 10-1 and on back of meter).
- 3. Multiply the saturation value by the altitude correction factor (Table 10-2 and on back of meter) to obtain a theoretical calibration value.

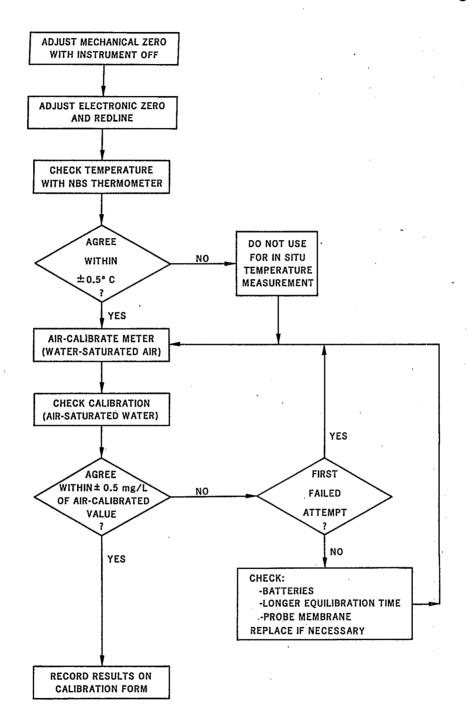


Figure 10-1. Flowchart for dissolved oxygen meter calibration.

Table 10-1. Solubility of Oxygen in Fresh Water<sup>a</sup>

Temperature (°C)	Dissolved Oxygen (mg/L)	Temperature (°C)	Dissolved Oxygen (mg/L
0	14.62	16	9.87
1	14.22	17	9.66
2 .	13.83	18	9.47
3	13.46	19	9.28
4	13.11	20	9.09
5	12.77	21	8.91
6	12.45	22	8.74
7	12.14	23	8.58
8	11.84	24	8.42
9	11.56	25	8.26
10	11.29	26	8.11
11	11.03	27	7.97
12	10.78	28	7.83
13	10.54	29	7.69
14	10.31	30	7.56
15	10.08	31	7.36

Reprinted from Standard Methods for the Examination of Water and Wastewater. 16th Edition, p. 413 (American Public Health Association, Washington, D.C., 1985).

Table 10-2. Altitude Correction Factors for Dissolved Oxygen Measurements

Atmospheric Pressure (mm Hg)	Equivalent Altitude (feet)	Equivalent Altitude (meters)	Correction Factor
775	-540	<b>-165</b>	1.02
760	0	0	1.00
745	542	165	0.98
730	1,094	333	0.96
714	1,388	423	0.94
699	2,274	693	0.92
684	2,864	873	0.90
669	3,466	1,056	0.88
654	4,082	1,244	0.86
638	4,756	1,450	0.84
623	5,403	1,647	0.82
608	6,065	1,849	0.80
593	6,744	2,056	0.78
578	7,440	2,268	0.76
562	8,204	2,500	0.74
547	<b>8,939</b>	2,725	0.72
532	9,694	2,955	0.70
517	10,472	3,192	0.68
502	11,273	3,436	0.66

- 4. Turn the selector switch to the appropriate range position.
- 5. Adjust the "CALIBRATE" knob until the meter reads the theoretical calibration value determined in step 3. Allow two minutes to verify the stability of the reading. Readjust if necessary.
- 6. Perform calibration check as described in Section 10.7.1.

NOTE: Calibration can be disturbed by physical shock, touching the membrane, or drying out of the electrolyte.

#### 10.5.2 Field Calibration

Prior to on-site measurements, the meter is recalibrated at streamside. The probe is placed into the calibration chamber (water removed), sealed, and placed into the stream for equilibration. Calibration follows the procedures presented in Section 10.5.1. The only difference is the use of the site altitude, obtained from appropriate topographic maps, and the lack of a post-calibration quality control check.

### 10.6 Procedure

The probe end should be protected by the screw cap and fully submerged in the water. The membrane end should not come in contact with the bottom, although the probe may lay on its side, with the end elevated off the substrate. Refer to Figure 10-2, Flowchart for field DO measurements.

- 1. Calibrate the meter using the air calibration procedure (Section 10.5.1).
- 2. Attach the probe to the sampling boom.
- 3. Immerse the probe in flowing stream water at middepth.
- 4. Turn the selector to "TEMP." Allow the reading to stabilize. Record water temperature on the field data form.
- 5. Turn the selector to the appropriate DO range. Allow the reading to stabilize. Record the DO reading on the field data form.

## 10.7 Quality Assurance and Quality Control

#### 10.7.1 Calibration Check

This quality control check is conducted after calibration at the base station, both before and following field activities. The QCC consists of comparing calibrated meter readings to the calculated DO of air-saturated deionized water, based on temperature and altitude. If measurements do not fall within limits for a QCC, recalibrate the meter (before field use) or qualify field DO measurements. If the meter cannot be calibrated so that it meets the QCC and a backup meter is not available, either qualify field data collected with the meter or correct the problem as described in Section 10.8.

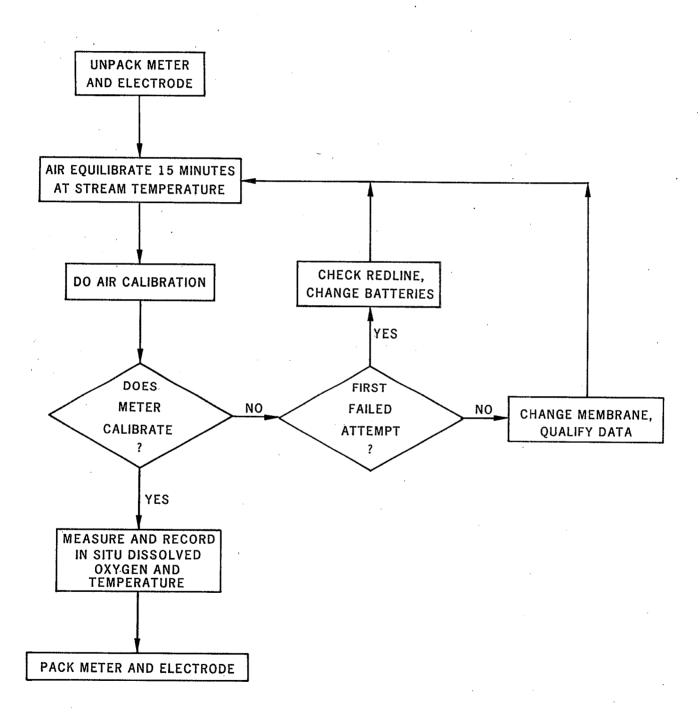


Figure 10-2. Flowchart for field dissolved oxygen measurement.

1. Air-saturate a bucket of deionized water by bubbling air through the water with an aquarium pump and air stone for a minimum of one hour (preferably several hours).

NOTE: Avoid large changes in ambient temperature during saturation.

- 2. Place the probe in the sample and stir gently. Set the selector to "TEMP," read the temperature of the bath, and determine the saturation value from the  $O_2$  solubility table (Table 10-1 and on back of meter).
- 3. Check the temperature reading using the following procedure:
  - a. Immerse the probe in a bucket of water.
  - b. Turn the selector knob to "TEMP." Check the real temperature on the meter against the temperature obtained by using an NBS-traceable thermometer. Readings should agree within 0.5 °C.
  - c. If the readings do not agree within 0.5 °C, do not use the probe to measure stream temperature. Instead, use the conductivity meter or an alternate method. This change should be noted on the field data form.
- 4. Determine the local altitude from a topographic map or obtain atmospheric pressure from a mercury barometer. Determine the atmospheric correction factor (Table 10-2 and on back of meter).
- 5. Multiply the saturation value by the atmospheric correction factor to obtain the theoretical dissolved oxygen concentration of the water.
- 6. Turn the selector to the appropriate DO range and take the DO reading while stirring the probe in the bucket. Calculate the difference between the calculated theoretical value and the measured values. The measured readings should be within ±0.5 mg/L O<sub>2</sub> of the calculated value. Record values on the calibration form and the field data form.
  - a. If the reading is outside of the acceptance limits, recalibrate with water-saturated air (Section 10.5) and repeat this calibration check.
  - b. If the reading is still not acceptable, check the probe and meter for malfunction.

## 10.7.2 Post-Deployment Calibration Check

After returning from the field, repeat the calibration and QCC procedures (Sections 10.5.1 and 10.7.1), perform maintenance, and/or troubleshoot the meter according to Section 10.8 and the manufacturer's instruction manual. Record information on the calibration form and, where appropriate, on the field data form.

## 10.8 Routine Maintenance and Care

Refer to the instrument manual for membrane replacement instructions.

1. Replace the membrane and electrolyte (or entire probe) if erratic readings are observed, if calibration is not stable, if bubbles form under the membrane, or if bacterial growth is

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observed on the membrane. Note especially the instructions concerning the pressure compensation diaphragm and removal of hidden bubbles.

- 2. If calibration is unstable after membrane replacement, let the membrane equilibrate for 24 hours. If the meter must be used during this period, data may have to be qualified with a comment such as "new membrane installed at site."
- 3. Check the meter and meter case frequently for moisture. The meter must be kept dry. Open the meter back and allow it to dry overnight if the meter is moist.

#### 10.9 References

American Public Health Association, American Water Works Association, and Water Pollution Control Foundation. 1985. Standard Methods for the Examination of Water and Wastewater. 16th Edition. American Public Health Association, Washington, D. C.

Yellow Springs Instrument Company. 1980. Instructions for YSI Model 54A meters. Yellow Springs Instrument Company, Yellow Springs, Ohio.

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# 11.0 Secchi Disk Transparency

### 11.1 Overview

The Secchi disk transparency measurement provides an in situ estimate of water clarity. This procedure requires no calibration and no quality assurance or quality control checks. Maintenance is limited to visual inspection for damage to the disk and sounding line.

### 11.2 Procedure

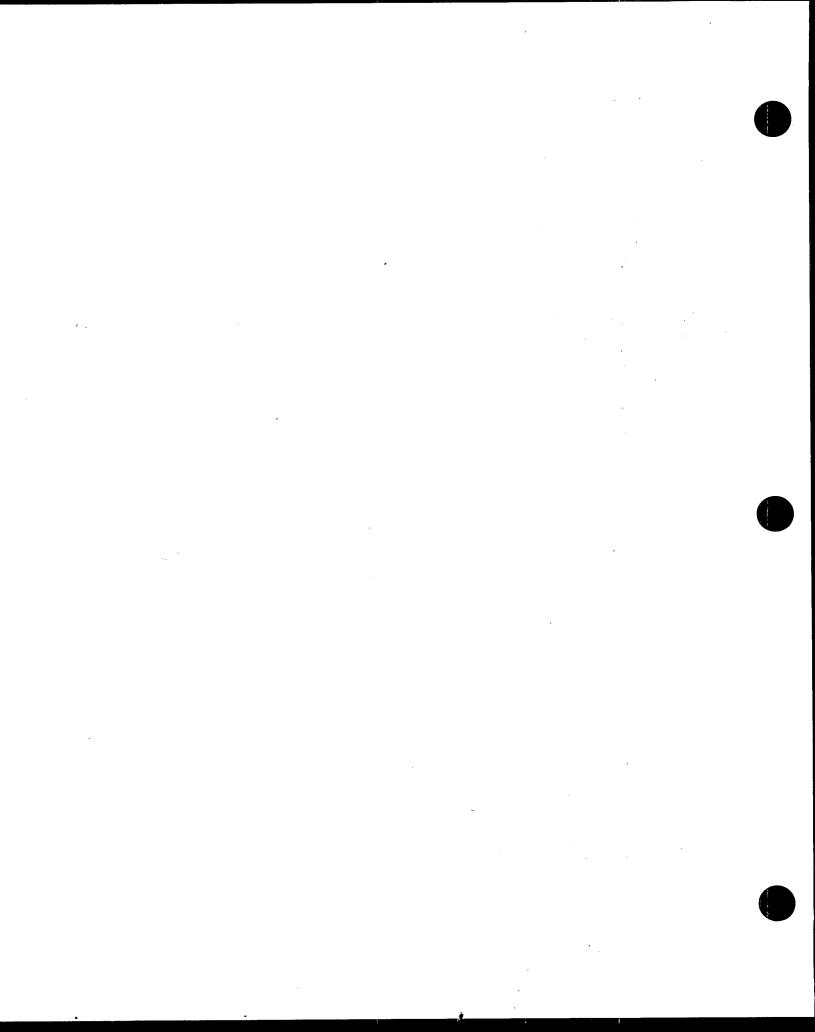
NOTE: Secchi disk transparency determination is made in the shade of the helicopter between the pontoon and fuselage or from the shaded side of the boat. If it is not possible to perform the measurement in the shade, make a note of this.

NOTE: The sampler must not wear sunglasses. Photogray prescription lenses are permissible if no other glasses are available. Their use should be noted.

1. Lower the Secchi disk on a calibrated line until it disappears from view. Record this depth on the field data form.

NOTE: Calibrated line refers to depth markings made against a standard tape measure.

- 2. Raise the disk until it reappears, then record this depth also.
- 3. The average of these depths is the Secchi disk transparency.



## 12.0 Water Sample Collection--Van Dorn Sampler

#### 12.1 Overview

These procedures are applicable to the collection of water samples from lakes of at least 1.5 m depth. Either helicopters or boats may be used as the sampling platform. The Van Dorn sampler was used as the primary water collection apparatus in all AERP lake surveys. All Van Dorn samplers used in the AERP Lake Surveys were fitted with nylon Leur-Lok syringe fittings to permit sample extraction without atmospheric contact.

This section describes the collection of standard lake water samples by using the Van Dorn sampler. These standard water samples include a 4-L bulk Cubitainer sample, syringe samples, and QA/QC samples. The collection of specialized seasonal samples are described in sections 14.0 through 17.0.

## 12.2 Water Sample Collection Procedure

- Set the sampler by pulling the elastic bands and cups back and securing the latches. Do
  not place hands inside or on the lip of the container; this could contaminate samples. To
  reduce chances of contamination, wear thin, sterile laboratory gloves.
- 2. Rinse the 6.2-L Van Dorn sampler with surface water by immersing it in the water column.
- 3. Lower the Van Dorn sampler to the desired sampling depth.
  - NOTE: Sampling depth may be 1.5 m below lake surface, 1.5 m off lake bottom, or some other depth as required by the survey design.
- 4. Trigger cups by releasing the messenger weight down the line.
- 5. Raise sampler and set on a clear, flat surface (helicopter pontoon or cooler lid) in a vertical position.
- 6. Extreme care must be taken to avoid leakage of sample and introduction of air.

## 12.3 Syringe Sample Collection Procedure

NOTE: For AERP lake surveys, syringe samples were collected (one each) for DIC, pH, extractable aluminum, and monomeric aluminum samples.

- 1. Unscrew valve at the top of the Van Dorn sampler. Remove plug from Leur-Lok syringe fitting at bottom of sampler.
- 2. Withdraw a 50-mL aliquot into the 60-mL syringe. Expel as waste (do not expel into Van Dorn). Repeat two more times.

- 3. Reattach syringe to the Van Dorn sampler with locking valve.
- 4. Withdraw a 60-mL aliquot, expel all air bubbles, and close valve on syringe.
- 5. Repeat steps 2 through 4 with additional syringes.
- 6. Attach completed labels (Appendix A, Figure A-10) to syringes; place syringes in plastic bag or plastic container; place in cooler.
- 7. Maintain at 4 °C with frozen gel packs.

# 12.4 Cubitainer Sample Collection Procedure

1. Label sides of Cubitainer by using a permanent waterproof marker. Pop out the mouth to expand Cubitainer size, using care not to touch the inner lip of the Cubitainer.

NOTE: Never expand Cubitainer by blowing into it; this could contaminate the sample.

- 2. Thoroughly rinse a clean 4-L Cubitainer with three separate 200-mL portions of sample. Cap and rotate so that the water contacts all surfaces. Discard each rinse.
- 3. Completely fill the Cubitainer with sample remaining in the Van Dorn sampler. If necessary, manually expand Cubitainer.
- 4. Compress Cubitainer to remove all headspace and cap it tightly. Tape clockwise with electrical tape.
- 5. Complete and attach field sample label to the Cubitainer.

NOTE: This information should duplicate the information written on the Cubitainer wall in step 1. The field sampler label should be attached to the Cubitainer neck with a rubber band.

6. Place sample in cooler with frozen gel packs.

## 12.5 QA/QC Samples

## 12.5.1 Duplicate Samples

NOTE: During AERP surveys, one duplicate sample was collected for each sample batch, defined as a group of samples processed in one day at an individual processing facility. Additional replicates were taken during specific surveys for analytical laboratory bias checks.

Immediately after collection of the routine sample, repeat sections 12.2 through 12.4. Mark all samples as duplicate samples. If additional replicate samples are taken, label as triplicate, etc.

### 12.5.2 Blank Samples

1. Rinse the Van Dorn sampler with 200 mL of deionized water three times.

NOTE: The deionized water should conform to ASTM specifications for Type 1 reagent grade water (ASTM, 1984).

- 2. Fill the Van Dorn sampler with deionized water.
- 3. Collect two syringe samples as described in Section 12.3.

NOTE: Blank syringe samples are not collected for pH and DIC analyses.

- 4. Complete and attach field sample label, identifying samples as blanks.
- 5. Thoroughly rinse a clean 4-L Cubitainer with three separate 200 mL portions of water from the sampler.
- 6. Rinse and fill a Cubitainer with the deionized water from the Van Dorn sampler (see Section 12.4). Compress the Cubitainer to remove headspace and cap it tightly. Tape clockwise with electrical tape.
- 7. Place the Cubitainer and syringe container in a cooler with frozen gel packs.

#### 12.6 References

ASTM (American Society for Testing and Materials). 1984. Annual Book of ASTM Standards, Volume 11.01, Standard Specification for Reagent Water, D 1193-77 (reapproved 1983). ASTM, Philadelphia, Pennsylvania.

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# 13.0 Water Sample Collection--Peristaltic Pump

#### 13.1 Overview

Water samples are collected from natural water bodies by use of a peristaltic pump and flexible tubing. The waters are directed into various sample containers for chemical analyses.

### 13.1.1 Scope and Application

This method is applicable to collection of waters from static and flowing waters. This procedure is similar to the one employed in the NSS, which utilized a 12-volt direct current (VDC) Masterflex Model 7533-30 pump motor with Quick Load head (Series 7020-50) and plastic Tygon tubing (Type R-3603) to collect stream water. The description presented here of this method assumes that the Masterflex peristaltic pump and accessories are used to collect flowing waters. The method is not limited to lotic systems. Collection of lake waters from different depths within the water column are possible with only few changes in this procedure. The method can also be used with modification for other instrumentation meeting equivalent specifications.

### 13.1.2 Summary of Method

The pump head, tubing, and power cables are attached to the pump motor. The end of the intake line (of appropriate length) is placed 20 to 30 cm below the stream surface and rinsed with pumped water for three to five minutes. Sample containers (e.g., 4-L Cubitainer, nalgene bottles, and syringes) are filled directly from water flowing out of the pump/tubing discharge. Syringes may be filled without exposing water to the atmosphere. All sample containers are rinsed three times prior to sample collection. Potential for cross-contamination is minimized by replacing all lines with unused tubing at each stream site.

## 13.2 Equipment and Supplies

- 1. Battery (12 VDC) and power cable.
- 2. Pump motor.
- 3. Pump head.
- 4. Tygon tubing (10 to 20 feet per sampling site).
- 5. Sample boom.
- 6. Plastic sheeting.

## 13.3 Preparation

Prepare the battery pack that will operate the pump daily. Standard Nicad batteries are charged 24 hours prior to use and discharged frequently to avoid memory. There are no routinely maintained components to the pump/head assembly. Refer to the pump and Quick Load head manual for cleaning and repair instructions. All sample containers and tubing should be prepackaged in sealed plastic bags to prevent contamination from road dust.

### 13.4 Assembly

Place the pump, battery, and sample containers on a sheet of clean plastic laid over a flat area adjacent to the stream. Connect the battery cable to the pump (positive and negative poles). Attach the pump head and insert the appropriate length tubing so that 12 to 18 inches remain on the output side of the head.

#### 13.5 Water Collection Procedure

- 1. Assemble peristaltic pump.
- 2. Attach length of tubing (1/4 inch ID) to pump and sampling boom. Tubing length may vary from 10 to 20 feet, as determined during site reconnaissance. Do not let tubing come in contact with the ground.
- 3. Affix completed label to all sample containers before sampling. Mark these labels as "Routine".
- 4. Immerse intake tubing into flowing water of a stream at middepth or at a depth of 20 to 30 cm.
- 5. Turn pump on. Purge tubing for two minutes.
- 6. Insert discharge tubing 3 cm into neck of empty 4-L Cubitainer.
- 7. Collect 20 to 50 mL of water in Cubitainer. Cap and rotate so that water contacts all surfaces. Discard water.
- 8. Repeat above rinsing procedure two more times.
- 9. Insert discharge tube into neck of Cubitainer and completely fill.
- 10. Cap Cubitainer tightly (no airspace should remain).
- 11. Rinse a 500-mL deionized-water-washed nalgene bottle as described above and completely fill (no airspace) for suspended solids analysis.

12. Collect 60-mL syringe samples (water unexposed to the atmosphere is necessary) as follows:

NOTE: For AERP lake surveys, syringe samples were collected (one each) for DIC, pH, extractable aluminum, and monomeric aluminum samples.

- a. Affix label so that graduations are visible.
- b. Fill a 60-mL syringe with stream water by placing syringe tip on end of discharge tubing and allow pump pressure to fill syringe. Expel water as rinse.
- c. Repeat rinse procedure two more times and then fill syringe with fresh sample.
- d. Affix syringe valve, tap side of syringe to collect air bubbles at tip, and then expel air. Close syringe valve.
- e. Repeat collection procedure above with three more syringes.

### 13.6 QA/QC Samples

#### 13.6.1 Duplicate Sample Collection

NOTE: During AERP surveys, one duplicate sample was collected for each sample batch, defined as a group of samples processed in one day at an individual processing facility. Additional replicates were taken during specific surveys for analytical laboratory bias checks.

A duplicate is collected completely independent of the routine sample and is taken to measure natural variation. After collecting the routine sample, repeat the procedure described in Section 13.5 with a second set of sample containers. Before collecting the duplicate sample, label these containers and mark them as duplicate samples.

#### 13.6.2 Field Blank Collection

A blank sample is deionized water, meeting ASTM specifications for Type 1 reagent grade water (ASTM, 1984), run through all field sampling gear; it is taken to measure potential field contamination. The sample is generally collected with the same tubing used for routine samples, but prior to actual routine water collection. Add all labels before sampling. Mark labels to indicate blank samples.

- 1. Immerse intake tubing into 4-L Cubitainer of deionized water.
- Purge tubing with at least 2 liters of deionized water. Discard the first liter of purge water.
  Use the remaining purge water to rinse sample Cubitainer, syringes, and suspended solids bottle. Rinse each container three times with approximately 20 to 50 mL of water.
- 3. Place rinsed 4-L Cubitainer under collection tubing and fill approximately half full with remaining deionized water in the first blank water Cubitainer.
- 4. Immerse intake tubing into second Cubitainer of deionized water and complete filling of Cubitainer.

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- 5. Eliminate airspace and cap tightly.
- 6. Fill rinsed suspended solids bottle with blank water, eliminate airspace, and cap tightly.
- Fill two syringes with blank water described in Section 13.5, step 12.
   NOTE: Syringe samples are not taken from blanks for DIC and pH measurements.

## 13.7 References

ASTM (American Society for Testing and Materials). 1984. Annual Book of ASTM Standards, Volume 11.01, Standard Specification for Reagent Water, D 1193-77 (reapproved 1983). ASTM, Philadelphia, Pennsylvania.

# 14.0 Nitrate/Sulfate Aliquot

#### 14.1 Overview

In addition to the standard Cubitainer and syringe water samples collected during NSWS, additional special interest samples were collected in some of the individual component surveys. These samples, which required immediate field treatment to preserve sample integrity, included a sample for nitrate/sulfate analysis. Other special interest samples are discussed in sections 15.0, 16.0, and 17.0.

### 14.1.1 Summary of Method

Nitrate is prone to rapid degradation in unpreserved, unrefrigerated samples. Preservation at the field site is recommended if the raw sample cannot be refrigerated at 4 °C immediately or cannot be processed by a laboratory within 24 hours of collection. During AERP surveys, the water sample was taken from the Van Dorn sampler; operation of the Van Dorn is described in Section 12.0. The sample was collected in a 125-mL opaque aliquot bottle and immediately preserved with 5 percent mercuric chloride (5% HgCl<sub>2</sub>). This procedure was used by ground samplers in the WLS.

### 14.1.2 Safety

In this procedure, 5% HgCl<sub>2</sub> is the preservative. Mercury is a hazardous material although, at the low concentration used, normal safety procedures for handling chemicals are generally adequate to ensure personnel safety. Gloves should be worn and all containers should be kept tightly capped when not in use. As an added precaution, it is recommended that personnel handling mercuric chloride receive analysis of body mercury levels both before and after the survey.

## 14.2 Equipment and Supplies

- 1. Van Dorn sampler.
- 2. 125-mL aliquot bottle, opaque polyethylene, one per sample.
- 3. 5% HgCl<sub>2</sub>.
- Eyedropper.
- Electrical tape.
- 6. Sample labels, one per sample.
- 7. Plastic bag (sandwich size, one per sample).
- 8. Frozen gel packs.
- 9. Cooler.

#### 14.3 Procedure

- 1. Rinse the sample bottle (125-mL, deionized-water-washed, amber, polyethylene bottle) with three separate 20-mL portions of sample from the Van Dorn sampler. Cap the bottle tightly each time and rotate the bottle to rinse all inner surfaces. Discard each rinse.
- 2. Fill the bottle to shoulder with sample from the Van Dorn sampler.
- 3. Using a dropper bottle, slowly add 2 drops (0.1 mL) of 5% HgCl<sub>2</sub> to the bottle. Note the amount of preservative used on the aliquot label. Cap the bottle tightly and invert it several times to mix.
- 4. Affix an aliquot label (Figure 14-1) and record all information, except laboratory-produced information (batch and sample ID) with an indelible marking pen.
- 5. Tape the bottle clockwise with electrical tape and place it in a plastic bag. Keep the samples at 4 °C.

NOTE: Bottle contraction may occur as a result of refrigeration. It may be necessary to retighten and retape bottle lids after 1 to 2 hours at 4 °C.

6. Repeat steps 1-5 for duplicate and blank samples.

	EMSL-LAS VEGAS SPLIT	
	Unfiltered-125 mL	
	Field Crew Data	
Lake ID		
Crew ID_ Sample Type (	check one)	
Rout		
	cut here	፱
Date Sampled		
Time Sampled Preservative:	HgCl <sub>2</sub> ml	
***	Field Lab Data	
Batch ID	Sample ID	
Parameters:	NO3, SO4	

Figure 14-1. Nitrate/sulfate aliquot label.

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JW	u	Amount:
	J. ≯ <sup>(6</sup> (	reservative; HNO
		OI dotsi OI alquis
	əmiT berətli—	balqmaS atac ami balqmas
	ANOXIC SPLIT 4 - Filtered - 125 mL	
	Sample Type	

Figure 15-1. Anoxic sample silquot label.

# 15.3 Preparation

### 15.3.1 Preparation of Filters

The following activity may be completed in the laboratory prior to survey commencement or in the field or at the base site prior to sampling. Approximately 4 or 5 filters are needed for each anoxic sample collected.

- 1. Fill a clean syringe with deionized water.
- 2. Westing gloves, attach a 0.45µm Acro-disk filter to the syringe. Inject syringe contents through the filter.
- 3. Remove the filter and place it in a sealable bag. Do not seal the bag opening.
- 4. Repeat steps 1-3 until a sufficient number of tilters have been rinsed. The same syringe and plastic bag may be used for numerous tilters.
- 5. Filters may be allowed to dry in the open bag inside a clean air station. Seal the bag when tilters are dry.

## 15.0 Anoxic Iron and Manganese Aliquot

## weiview 15.1

In addition to the standard Cubitainer and syringe water samples collected during NSWS, additional special interest samples were collected in some of the individual component surveys. These samples, which required immediate field treatment to preserve sample integrity, included a sample for anoxic iron and manganese analysis. Other special interest samples are discussed in sections 14.0, 16.0, and 17.0.

There is a concern about the potential loss of dissolved iron and manganese in water samples collected from the hypolimnetic zone of stratified lakes resulting from oxidation processes during collection. To reduce the potential risk of oxidation, a field-preserved aliquot should be prepared from samples collected at the mid-hypolimnion in stratified lakes or from 1.5 m above the bottom in isothermal lakes. This aliquot should be collected in a sealed syringe to avoid exposure to atmospheric oxygen and is filtered and preserved immediately after collection. Anoxic samples should be collected only during seasons in which the hypolimnetic zone is expected to be oxygen depleted (e.g., during periods of strong stratification).

## 15.2 Equipment and Reagents

The following items are required to collect and prepare anoxic samples:

- 1. Disposable syringe with syringe valve, one per sample.
- 2. Syringe filters (disposable), four to five per sample.
- 3. Aliquot label (see Figure 15-1), one per sample.
- 4. 125-mL acid-washed aliquot bottle with 0.2 mL Ultrex nitric acid, one per sample.
- 2. Indelible marking pen, two per team.
- 6. Disposable gloves (powder-free), one pair per sample.
- 7. Electrical tape, one roll per team.
- 8. Plastic bag (sealable sandwich type), one per sample.

# 15.3.2 Preparation of Aliquot Bottles

NOTE: Due to potential hazards associated with handling concentrated acids in the field, this activity should be performed in the laboratory. One bottle is needed for each anoxic sample.

1. Attach a clean micropipet tip to a calibrated 40-200 $\mu$  micropipet. Wearing safety glasses, lab coat, and disposable gloves, pipet 0.2 mL of Ultrex nitric acid into a clean 125-mL acid-washed, polyethylene aliquot bottle. The same pipet tip may be used for several bottles if care is taken not to touch any surface.

NOTE: Clear aliquot bottles are preferred over amber bottles due to the potential of iron leaching by the concentrated acids.

Cap the aliquot bottle tightly. Tape the bottle clockwise with electrical tape and seal it in a plastic bag.

## 15.4 Procedure

- Collect a bulk water sample in a Van Dorn from the mid-hypolimnion or from 1.5 m above the bottom (see Section 12.0 for operation of the Van Dorn sampler).
- Affix a syringe to the syringe port on the Van Dorn, and draw rinse water into the syringe three times. Fill the syringe with sample. Avoid introducing air into the syringe during sample collection.

NOTE: The Van Dorn should be positioned with the syringe port on the bottom. Anoxic samples are collected first, prior to collection of any other water samples.

 Attach a rinsed Acro-disk filter. Eject 5 to 10 mL through the filter; discard as waste. Inject the remaining sample through the filter, into a 125-mL aliquot bottle containing 0.2 mL nitric acid.

CAUTION: Injection into the acid-spiked bottle may cause fuming or splashing. Samplers should keep their heads far away from the bottle opening and wear double gloves.

If the filter clogs, discard the filter, and proceed from the beginning of this step with a new filter.

- 4. Repeat steps 2 and 3. The total sample volume should be 70 mL (minimum) to 110 mL (maximum). The same syringe may be used for both filtrations of one sample; however a new syringe should be used for each new sample.
- Cap aliquot bottle, wrap clockwise with electrical tape. Seal in plastic bag and store at 4 °C.
- 6. Repeat from step 1 for any duplicates or blank samples.

## 16.0 Chlorophyll a Aliquot

### 16.1 Overview

In addition to the standard Cubitainer and syringe water samples collected during NSWS, additional special interest samples were collected in some of the individual component surveys. These samples, which required immediate field treatment to preserve sample integrity, included a sample for chlorophyll  $\alpha$  aliquot analysis. Other special interest samples are discussed in sections 14.0, 15.0, and 17.0.

Chlorophyll  $\alpha$  is one of several chlorophylls found in planktonic algae and is commonly measured as an indicator of algal biomass. Chlorophyll  $\alpha$  deteriorates rapidly after collection; therefore, field filtration and immediate freezing are required.

## 16.2 Equipment and Supplies

- 1. Van Dorn sampler.
- 2. 2-L amber widemouth polyethylene container or other suitable opaque sample container.
- 3. Filtration apparatus, hand operated.
- 4. Polycarbonate filter, 0.8μm pore size (two per sample).
- 5. Graduated cylinder, 250 mL.
- Deionized water and wash bottle. The deionized water should conform to ASTM specifications for Type 1 reagent grade water (ASTM, 1984).
- 7. Forceps.
- 8. Centrifuge tube, 10-mL polycarbonate with screw-cap.
- 9. Aliquot label (see Figure 16-1), one per sample.
- 10. Sealable plastic bags.
- 11. Electrical tape.
- 12. Frozen gel packs or dry ice.

Lake ID-	, and the second
Crew Sample Type	
Date Sampled	Time
Volume Filtered	j m
Batch ID	III E
Sample ID	
Preservative: - Parameter: Ch	20 °C Norophyll

Figure 16-1. Chlorophyll  $\alpha$  aliquot label.

### 16.3 Procedure

## 16.3.1 Preparation and Sample Collection

- 1. Load filter holder with a filter. Rinse thoroughly with deionized water. This may be done prior to going to the field. If so, the filter holder should be transported in a sealable plastic bag. A second filter should be taken as a back-up.
- 2. Thoroughly rinse graduated cylinder and sample bottle with deionized water.
- 3. After collection of all other water samples, gently agitate the residual sample in the Van Dorn sampler and decant into sample container. Label container and store at 4 °C.

NOTE: Storage time should be as short as possible. If sampling from a boat, sample may be stored until shore is reached. Helicopter samplers should filter sample immediately.

#### 16.3.2 Filtration

NOTE: Chlorophyll can degrade rapidly when exposed to bright light. The entire filtration procedure *must* be performed in subdued light. Centrifuge tubes containing sample filters must also be kept in subdued light.

1. Gently invert the sample container three times and decant exactly 250 mL of sample into the graduated cylinder.

NOTE: The volume must be exact for later use in analytical calculations. If sample size is less than 250 mL, record actual volume to the nearest mL.

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- 2. Pour sample into the top of the filter holder, replace holder cap and pump sample through the filter using the hand pump. Filtration pressure should not exceed 7 psi to avoid rupture of fragile algal cells.
- Thoroughly rinse the upper portion of the filtration apparatus with deionized water to dislodge any remaining cells adhering to the sides. Check the volume of the lower chamber, which traps the filtrate, to ensure that it does not make contact with the filter membrane.
- 4. Remove the filter from the holder with clean forceps. Avoid touching the filter where the algal cells are deposited. Fold the filter in half, then into quarters, and insert into a screw-cap, 10 mL centrifuge tube. Place the tube inside a sealable plastic bag and tape to the underside of a frozen gel pack. Sandwich the sample between two gel packs, and store inside a cooler beneath the Cubitainers and syringes. Transfer to a -20 °C (minimum) freezer as soon as possible.

NOTE: Filters should be frozen immediately, and kept frozen until analysis can be performed. Severe deterioration can occur under varying temperature conditions.

#### 16.4 References

ASTM (American Society for Testing and Materials). 1984. Annual Book of ASTM Standards, Volume 11.01, Standard Specification for Reagent Water, D 1193-77 (reapproved 1983). ASTM, Philadelphia, Pennsylvania.

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# 17.0 Collection and Preservation of Zooplankton

#### 17.1 Overview

In addition to the standard Cubitainer and syringe water samples collected during NSWS, additional special interest samples were collected in some of the individual component surveys. These samples, which required immediate field treatment to preserve sample integrity, included zooplankton samples. Other special interest samples are discussed in sections 14.0 through 16.0.

The following procedures describe the collection and preservation of zooplankton samples. Identification and species counts provide an estimate of zooplankton community composition, an important indicator of biological health of an aquatic ecosystem. The procedures presented here were developed for and employed during the ELS-II summer seasonal survey.

#### 17.1.1 Scope and Application

These procedures are most applicable to lakes of 3 m or more in depth. Generally these procedures should not be used in lakes of less than 1.5 m in depth. Additionally, these procedures are recommended only for use in seasons of zooplankton productivity.

### 17.1.2 Summary of Method

Three vertical tows are taken at the deepest part of the lake, from 1.5 m above the bottom to the surface. Collected matter is transferred from the collection net to a sample container and immediately preserved with a buffered formalin-sucrose solution. Preserved samples may be stored indefinitely for subsequent identification and count of zooplankton species. Zooplankton collected by this procedure retain body shape. This facilitates subsequent identification.

# 17.1.3 Safety

Formalin is considered a hazardous material and should be treated as such. The solution and all samples containing the preservative must be stored separately from all other samples. While handling formalin, personnel should wear gloves and eye protection and avoid respiration of fumes (mechanical respiratory protection is not required). Formalin is a restricted article; therefore shipments should be made only by personnel and carriers trained and *authorized* to do restricted article shipping. A toxic substance safety plan should be prepared.

# 17.2 Equipment and Supplies

- 1. Wisconsin Style plankton net, 80  $\mu$ m mesh.
- 2. Sample containers, 250-mL, widemouth glass jars with screw lids.

- 3. Deionized water, meeting ASTM specifications for Type 1 reagent grade water (ASTM, 1984).
- 4. Formalin solution, prepared as follows:
  - a. Dilute formaldehyde in a 1:1 volume ratio with deionized water.
  - b. Add 3 g of Borax per liter of formalin; the pH of the solution should be pH 7.5-8.0.
  - c. Add an odor reducer as per instructions on label (optional).
  - d. Rinse clean containers three times with small (10 mL) portions of the formalin mixture; discard rinses. Fill each bottle and cap tightly. Tape the lids clockwise with electrical tape. Label the bottles "Formalin." One-liter Tox bottles (Teflon-wrapped glass) or two-liter opaque polyethylene bottles may be used.
    - CAUTION: Due to safety concerns, steps 1 through 4 should be conducted in a true fume hood, (not a clean air station), by an analyst wearing a lab coat, safety glasses, Viton gloves, and a (optional) respirator.
  - e. Store the formalin in a cool place. Refrigeration is desired but not necessary.
  - f. Before use, add sucrose in a 20 percent weight:volume ratio.

NOTE: Formalin must be kept cool once sucrose has been added.

#### 17.3 Procedure

1. From the boat stern, lower the plankton net to 1.5 m above the bottom of the lake. Record depth.

NOTE: If lake is less than 3 m deep, carefully drop the net to the lake bottom.

- 2. Pull the net upward at a constant and moderate pace (not less than 10 m/minute) until the net reaches the surface.
- 3. Thoroughly rinse the net by splashing lake water through the sides so that all particulate matter is rinsed down into the sampling bucket. Care should be taken to ensure that splash water is not allowed to enter the net through the open mouth. Visually inspect the netting to ensure all particulate matter has been washed down into the sampling bucket.
- 4. Without loosening the drain stopper within the sampling bucket, carefully disconnect the basket from the net. Place the drain hole above a 250-mL glass sample jar. Remove the drain stopper. With a wash bottle containing deionized water, rinse all the particulate matter in the sampling bucket into the sample jar. Final volume should be approximately 200 mL.
- 5. Add 50 mL of 50 percent formalin (prepared as described in Section 17.2) to the sample to yield a final concentration of approximately 10 percent formalin and 4 percent sucrose.

- 6. Cap and label (Figure 17-1) bottle. Tape cap clockwise with electrical tape and place in plastic bag. Keep separate from other water samples. Samples do not require refrigeration, but should be stored in a cool place.
- 7. Repeat the above procedure from the side of the boat and a third time from the bow.

Lake ID	
Crew	
Date Sampled	
Time Sampled	
Depth	meters
Tow No:of	
Batch ID	
Sample ID	
Preservative: Formalin	
Parameters: Zooplankton	

Figure 17-1. Zooplankton sample label.

# 17.4 Quality Assurance/Quality Control

No QA or QC procedures are applicable to the collection procedures. Due to the spatial distribution variability of zooplankton within a lake, the replicate tows should not be considered as true QA duplicates.

#### 17.5 References

ASTM (American Society for Testing and Materials). 1984. Annual Book of ASTM Standards, Volume 11.01, Standard Specification for Reagent Water, D 1193-77 (reapproved 1983). ASTM, Philadelphia, Pennsylvania.

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# Appendix A

# National Surface Water Survey Blank Data Forms

The National Surface Water Survey forms shown in this appendix are facsimiles of the forms used in field operations.

Figure Number	Form Title	<u>Page</u>
A-1	Hydrolab calibration form	2 of 12
A-2	Lake coordinates form	3 of 12
A-3	Daily itinerary form	4 of 12
A-4	Field communication sheet	5 of 12
A-5	Incoming telephone record	6 of 12
A-6	Lake data form	7 of 12
A-7	Watershed characteristics form	8 of 12
A-8	Stream data form	9 of 12
A-9	Hydrologic data form	10 of 12
A-10	Field sample label	11 of 12
A-11	Flight plan	12 of 12

	HYD	NATIO ROLAI	NAI 3 S	SURF URVEY	ACE OR	WATI	ER SUR	VEY ON FO	ORM		
METER IO:		CR	ΕW	ID:			NAME:				
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							RRECTED	)			
	DAT	ΓE	1	ГІМЕ		BARO	METRIC SURE (mr			V	OLTAGE
PRE-CAL											
POST-CAL					•						
				pH CALIB	RATI	ON CHE	ск				
-	TEMP	(° C)		THEOR. VALUE		TIAL	SULDA NY	TED	FINA	L	RECAL
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4.00 BUFFER											
7.00 BUFFER							If to	Y, go Recal.			
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							ON CHEC ADJUS				
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CAL SAVED?_											
		DO	CAL	BRATION	N CHE	ECK (IF	APPLICA	BLE)			
	TEMP	(° C)	Ţ	THEOR. VALUE	IN	IITIAL	ADJUS Y/I	TED 1	FINA	<u>L</u>	
PRE-CAL		-									
POST-CAL											
CAL SAVED?											-
		CO.	QUA	LITY CO	NTRO	L CHEC	CK SOLU	LION			
		_		DEPLOYM			POST D		YMENT		
		THEO	R.	METER	₹	DIFF.	THEO	R.	METER		DIFF.
TEMP. (+/- 1° (	<b>5)</b>	NBS			Т		NBS			T	**
pH (+/- 0.15)		*			十		*	_		†	
COND. (+/-20	μS/cm)	*			1.		*			T	
COMMENTS:											
* Table 7-2	2, Surve	yor II pr	oce	dures							······································
** Table 7-1											

Figure A-1. Hydrolab calibration form.

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			DATE	
TEAM		CREW		WEIGHT
		PILOT		,
			TOTAL WEIGHT	
LAKE 1.D.	LATITUDE	LONGITUDE	NAME	
	xx° xx.xx'	xx° xx.xx¹		
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TEAM I.D.:	APPRO CHECK	XIMATE -IN TIME:
VEHICLE DESCRIPTION:	ESTIMA	
LICENSE NO.:		
	SAMPLER #1	SAMPLER #2
NAME:		
CLOTHING:		
_		
DESTINATIONS:		
ROUTES OF TRAVEL:		
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#### FIELD COMMUNICATION SHEET

Date: / /		Base Site:	**····
Time:		Caller Name:	
* *	,	Receiver Name: _	
Sampling Summary:		•	
lumber of Lakes visited:			
Samı	ole		
AKE ID + Typ	e	COMMENTS	_
		•	1 2
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3			
4			
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5	<del></del>		
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7			g
8	4.		
9			
0			
egend:			
Sample Type: R=Routine; D=Duplica	te; B=Blank		
SHIPPING SUMMARY: (TO LAS VEGA	NS)		
Number of Syringes:	Shipped	Via: Fed Ex	Other
lumber of Cubitainers:	Airbill #:		
lumber of Shipping Coolers:	Saturday	/ Delivery: □	
light Information:			
Airline Flight # Orig	ination Dep.	Destination	Arrival
		Destination	Aniva
<del></del>	·		
		<del></del>	
Veather:		Next Day's Proje	ection:
·		•	
Audit Samples Requested:			
Batch ID:			Verified
Date Received:	(In Las Vegas)	Date: .	/

Figure A-4. Field communication sheet.

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INTERNAL COMMUNICATION SHEET - NSWS	- INCOMING TELEPHONE RECORD
DATE OF CALL: TIME OF CALL:	LOCATION:
CALLER NAME: RECEIVER	NAME:
PURPOSE REQUEST NUMBER REQUEST RN-	CORRECTIVE ACTION  NOTIFIED WAREHOUSE (Y,N)
(1)	
(3)	
(4)	
(5)	
(6)	
(1)	
(2)	
(4)	
(5)	
(6)	
(1)	
(2)	
(4)	
(5)	
(6)	

NATIONAL SURFACE WATER SURVEY				
	LAKE DATA FORM 1D	Page of (Total Shipment)		
METEOROLOGICAL DATA Air Temp. + / - ° C (circle)  ST. WIND SPEED:	Lake I.D.  Lake Name  D D M M M YY  ACCESS HELICOPTER HELICOPTER OTHER OTHER SITE DEPTH:  (It) X 0.3048m/It = m O  SECCHI DEPTH: Visible To Bottom	FIELD CREW DATA SIGNATURE  / Sampler / OC sign.  Hydrolab Quality Control Data Meter ID: OCCC CABLE SOMOE Initial: PH O Initial: PH O Final: Final: PH O Final: PH 2SO <sub>4</sub> (pH 4.00) Final: PH 2SO <sub>4</sub> (pH 4.00) FIELD QC TOLERANCES PH = ±0.20 Conductivity = ±20.0 µS/cm		
(Quadrangle Name and State)	°C D.O. IFZ IFA	3 10OO 3 15OO 5 20OO 2 25OO 4 30OO 6 35OO		
COMMENTS  FIELD NOTES: (NOT FO	DR KEYPUNCH)	Data Qualifiers  (A) Instrument Unstable  (D) Stow Stabilization  (D) Did Not Meet QCC  (C) (D) (D) Other (explain in Comment section)  FIELD LAB USE  TRAILER ID  BATCH ID		

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GILL'S (702) 362-2100

#### **NATIONAL SURFACE WATER SURVEY WATERSHED CHARACTERISTICS** DDMMMYY FORM 7 DATE U/L STREAM NAME LATITUDE: STREAM ID LONGITUDE: □ (ft) □ (m) ELEVATION: COUNTY STATE 1:250,000 MAP NAME MAP DATE STREAM WIDTH (m) 1:24,000 MAP NAME MAP DATE STREAM DEPTH (m) WATERSHED ACTIVITIES/DISTURBANCES BANK COVERAGE WITHIN 100 METERS OF (Check all that apply) STREAM BED (Check all that apply) Туре Distance From Stream (meters) ☐ Roadways Along Stream: **Deciduous Trees:** Paved Unpaved Coniferous Trees: ☐ Crossings Above Stream: ☐ Culvert☐ Bridged☐ Grade Wetland Areas: Grasses and Forbs: D Dwellings: Moss: Single Multiple . 🗖 Rocky/Bare Slopes: ☐ Agriculture: ☐ Cropland ☐ Pasture ☐ Fenced ☐ Unfenced 0 STREAM SUBSTRATE (Check all that apply) ☐ Industry: Heavy >75% Туре Logging. Approx. Age: Fires. Approx. Age: $\Box$ Boulders: > 25 cm ☐ Mine/Quarry Type: Cobble: 6-25 cm ☐ Impoundments, Type: . ☐ Above Site ☐ Below Site Gravel: 0.2-6 cm Sand: < 0.2 cm ☐ Livestock, Type: Ò Silt and Clay: O Other Aufwuchs: **PHOTOGRAPHS COMMENTS:** FRAME ID AZIMUTH LAP CARD FIELD CREW DATA **DATA QUALIFIERS** FORM DISTRIBUTION White Copy — ORNL Pink Copy — EMSL-LV Yellow Copy — FIELD CREW ID . **8**8 SAMPLER 1 SAMPLER 2 Revised 1-86

Figure A-7. Watershed characteristics form.

SAMPLER 3

CHECKED BY

NATIONAL SURFAC STREAN FOR	SHIPPING INFORMATION	
STREAM ID: STREAM NAME:	U/L ELEVATION: PHASE I VISIT #:	D D M M M Y Y  DATE SHIPPED:
SAMPLE DATE: D D M M M Y Y  PROGRAM: SAMPLES COLLECTED  PHASE I PROUTINE SCREENING DUPLICATE EPISODE PILOT BLANK	EPISODE SAMPLE TYPE  Base Flow - EPISODE ONLY  Base Flow - EPISODE AND PHASE I  RISING STAGE  PEAK STAGE  FALLING STAGE	TO:
TIME GAUGE HEIGHT (ft)  START: O  FINISH: O	RAIN CLOUD COVER  (CHECK ONE ONLY)  ID NO ID PREV ID MOD ID LIGHT ID HEAVY	# OF COOLERS  TOTAL # OF SAMPLES  # OF SAMPLES THIS  GOOLER
PH Y N (FIELD RECALIBRATION?) □ □ QCCS = pH 4.00	UNCOMPENSATED CONDUCTIVITY uS cm-1	DISSOLVED OXYGEN mg / I QCC = Theoretical — Measured
QCCS INITIAL:	QCCS INITIAL:	INITIAL:

COOLER TEMPERATURE  AT SHIPMENT ON RECEIPT  COCOCOCOCOCOCOCOCOCOCOCOCOCOCOCOCOCOC	NOT SAMPLED  ☐ INACCESSIBLE ☐ NO ACCESS PERMIT ☐ TOO SHALLOW ☐ COND. > 500 uS/cm ☐ pH<3.30	DATA QUALIFIERS  A INSTRUMENT UNSTABLE  D SLOW STABILIZATION  Q DID NOT MEET QCC  X  Y  Z
☐ BLANK SAMPLE ID	FIELD CREW DATA	FORM DISTRIBUTION
EPISODE SAMPLES ID:  □ BASE	SAMPLER 1	WHITE COPY — ORNL PINK COPY — EMSL-LV YELLOW COPY — FIELD ORANGE COPY — MOBILE LAB
☐ PEAK	SAMPLER 3CHECKED BY	Revised 1-6-86 GILL'S (702) 362-2100

Figure A-8. Stream data form.

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	CE WATER SU GIC DATA M 4A	JRVEY	SHEE DATE FLOW	D :	D —— ER I	o M  D:	М	M	Y	Y
STREAM ID:	'	SAMPLE I PHASE I  SCREENING EPISODE P	3	EPISC BAS BAS RIS PEA FAL	SE - E SE - E ING AK	PISO PISO	DE ON	NLY		_
TIME START::  TIME END::  MEASURED HYDROLOGY:	DEPTH (maxft.) WIDTH (meters) VELOCITY (m sec -1)	·	_0 _0 _0		ST.	. M	IEAS.			
TIME: STA	GE (ft) STEEL ROD	STAGE (ft.) O O		WID7	ΓΗ (r •-	n) C	)			
INTERVAL CENTER (m)  1.	DEPTH AT CENTER (II) CENTER (III) CENTER (II	VELOCITY AT NTER (m sec	<u>~</u> 000000000000000							
FIELD CREW DATA:  CREW ID:  SAMPLER 1:  SAMPLER 2:  SAMPLER 3:  CHECKED BY:	DATA QUALIFIERS  (a) INSTRUMENT UNSTABLE (b) SLOW STABILIZATION (c) OID NOT MEET QCC (c) (c) (c) (c) (c) (c) (c) (c) (d) (d) (d) (d) (d) (d) (d) (d) (d) (d	-E				PIN	HITE C K COI LLOW Rev	ISTRII OPY	– ORI EMSL - FIE -86	NL -LV

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LAKE ID	CREW ID
DATE SAMPLED	TIME SAMPLED
SAMPLE (Check	
ROUTINE DUPLICATE BLANK	
BATCH ID	SAMPLE ID

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	<u>FL</u>	IGHT PLAN	
DATE			
A/C No		A/C COLOR	
COMPANY NAME			PHONE
PILOT'S NAME			
PAX			WT
FUEL ON BOARD			W1
ROUTE OF FLIGHT:			
PROPOSED FUEL STOPS:			
PROPOSED FUEL STOPS:			
ACTUAL T/O TIME:	LOCATION	CHECK-IN <sub>.</sub>	TIME LOCATION
ACTUAL T/O TIME: TIME PROPOSED	LOCATION	CHECK-IN ACTUAL —	p
ACTUAL T/O TIME:  TIME PROPOSED	LOCATION	CHECK-IN ACTUAL ACTUAL	
TIME PROPOSED PROPOSED	LOCATION	CHECK-IN ACTUAL ACTUAL ACTUAL ACTUAL	
TIME PROPOSED PROPOSED	LOCATION	CHECK-IN ACTUAL ACTUAL ACTUAL ACTUAL	
TIME PROPOSED PROPOSED	LOCATION	CHECK-IN  ACTUAL  ACTUAL  ACTUAL  ACTUAL  ACTUAL	
TIME PROPOSED PROPOSED PROPOSED	LOCATION	CHECK-IN  ACTUAL  ACTUAL  ACTUAL  ACTUAL  ACTUAL	
TIME PROPOSED PROPOSED PROPOSED	LOCATION	CHECK-IN  ACTUAL  ACTUAL  ACTUAL  ACTUAL  ACTUAL	

# Appendix B

# Helicopter Safety Guidelines

# **B.1 General Responsibilities**

#### **B.1.1 Responsibilities of Passengers**

Passengers should pack equipment and supplies safely to avoid problems during flight. The following items should be included:

- 1. Clothing for the weather expected and the activities planned at the destination.
- 2. Medication for motion sickness, if needed. Those who are susceptible to motion sickness may need to take preventative medication. Anyone afflicted with acrophobia may also have problems as a passenger.
- 3. Survival gear needed for rugged, remote terrain and inclement weather.

#### B.1.2 Responsibilities of Pilot

The pilot is responsible for the safety of the aircraft at all times. Before each flight, the pilot checks fuel supply and inspects the aircraft carefully. The pilot also inspects the radio, compass, and other navigation equipment and makes sure all cargo is properly secured. Completion of a weight and balance plan is an FAA safety requirement.

The pilot's other responsibilities include the following activities:

- Before embarking, the pilot should always check current and forecasted weather conditions along the flight route and at the destination. Detailed weather information can be obtained at the time the pilot files the flight plan with the FAA Flight Service Station.
- 2. The pilot uses the weather information for plotting the route of flight, based on the performance characteristics of the aircraft that will be used.
- 3. If weather conditions are unfavorable, the pilot may decide to postpone the trip. Two types of weather that adversely affect flying are high winds and fog. Near large bodies of water and in coastal areas, fog is the most common and persistent weather hazard. A few degrees of change in temperature can cause fog to form rapidly over a wide area, making it dangerous to navigate and to land.
- 4. Helicopters have limitations related to weather conditions and maximum wind speeds. Plan survey activities so that the limitations are not exceeded.

5. The operation of helicopters is normally limited to daylight hours. Daylight hours are defined as one-half hour before sunrise and one-half hour after sunset.

#### B.1.3 Helicopter Sampling Personnel Responsibilities

#### B.1.3.1 Check-in Procedures--

- Helicopter personnel should check in with the base site at scheduled intervals during the day. Helicopter personnel should provide the base coordinator with an estimated time of arrival, updated through the day as necessary.
- 2. If the estimated time of arrival is changed because of wind, weather, sampling difficulties or other problems, it is important that the pilot notify the nearest Flight Service Station so that search and rescue procedures are not initiated unnecessarily.

#### B.1.3.2 Search and Rescue--

- 1. A search along the sampling route filed in the flight plan is initiated by ground crew personnel.
- 2. If this fails to locate missing personnel, federal, state, and local authorities, as appropriate, should be notified.

### **B.2** Flight Plans

Flight plans are extremely important to the safety of any flight. A suggested flight plan format is depicted in Appendix A, Figure A-11.

- 1. A flight plan is recorded on a simple form that is completed after the flight arrives at its destination.
- 2. The reverse side of the flight plan has a preflight checklist with space for recording information such as enroute weather, weather advisories, weather at the destination, and winds aloft.
- 3. Flight plans are filed by the pilot with the local FAA Flight Service Station. The pilot is also responsible for reporting any changes in flight plans and for reporting arrival at the destination, which completes the flight plan.
- 4. The flight plan provides the basic information necessary to search for the aircraft if it is delayed and does not reach its destination within a short period after your estimated time of arrival.
- 5. If the aircraft has flight difficulty and has to make a forced landing and the pilot and passengers were not able to call for help, a series of search procedures must be taken to locate the aircraft.

# **B.3 Safety Equipment**

- 1. A flight helmet (equipped with radio headphones) provides hearing and impact protection and allows communication among personnel while aboard the helicopter.
- 2. A Nomex flight suit provides some protection against fire and hypothermia.
- 3. A safety harness prevents falls from the helicopter while sampling.
- 4. Life vests or personal flotation devices are required for each person on board.
- 5. Fire-retardant gloves, constructed of Nomex and leather, must be supplied.
- 6. Leather boots should be worn.

#### **B.4 Ground Operations**

#### B.4.1 Preparing and Loading Equipment and Materials

- 1. To avoid chemical damage to or contamination of aircraft, chemicals and samples must be carefully packaged.
- 2. Each item of field equipment and each box of material should be weighed and marked with its weight before it is packed on the aircraft. This allows the pilot to calculate the weight and balance plan.
- 3. The cargo should be placed in locations designated by the pilot and tied down securely.
- 4. The chin section of the helicopter, located directly in front of the front passenger's feet, consists of a thin, clear plastic material. Do not place or drop anything in this area.

# B.4.2 Approaching the Aircraft

- 1. Since propellers and rotors are often difficult to see and avoid, especially when they are rotating, there are important precautions that should be followed:
  - a. Always keep clear of helicopter rotors.
  - b. Approach any aircraft in view of the pilot, so you can be seen before the pilot starts engines or moves the aircraft.
  - c. Stay at least 100 feet from helicopters at all times unless required to go nearer.
  - d. Keep clear of the tail boom of a helicopter and avoid walking under it or anywhere near the tail rotor blades.
  - e. Approach a helicopter on the same level as the helicopter. If you approach from a higher level than where the helicopter is standing or hovering, you may be dangerously close to the blades.

- f. Walk when approaching or leaving a helicopter; move in a crouch because the main rotor blades can be blown below their normal operating level.
- g. Whenever rotors are turning on a helicopter, passengers, pilots, and crew members should wear protective helmets.
- h. Goggles should be worn by all personnel who load, service, fuel, or fly in helicopters to prevent eye injury from dust and dirt stirred up by the rotors.
- Hearing protection should be worn when working around helicopters to prevent hearing loss

#### **B.4.3 Landing Areas**

- 1. Safe use of landing areas requires certain precautions and safety measures. Smoking regulations should be enforced at all landing areas.
- 2. Landing areas should be equipped with adequate fire extinguishers for possible emergency use during landing and takeoff. Several large dry chemical or foam fire extinguishers should be available.
- 3. Ground vehicles should not be moved near an aircraft until its rotors or propellers have stopped.
- 4. Unpaved helicopter landing and refueling areas should be swept or wetted down to prevent gravel or dust from being blown about. Landing areas should be kept clean.

#### B.4.4 Refueling

The following precautions should be taken before aircraft are refueled:

- 1. A fire extinguisher should be available.
- 2. The fuel tank or fuel truck and the aircraft should be electrically grounded.
- 3. The engine should be shut off and propellers or rotor blades stopped.
- 4. There should be no passengers on board the aircraft.
- 5. No unauthorized persons should remain within the refueling area.
- 6. No smoking should be allowed within 100 feet of the refueling operation.

Training of ground personnel should include:

- 1. Review of standard procedures.
- 2. Review of notification procedures.
- 3. Practice in emergency fire fighting and first-aid procedures.

Safety considerations in ground operations include:

- 1. Ways of approaching the aircraft.
- 2. Fire fighting preparations.
- 3. Refueling precautions.

### **B.5 In-Flight Precautions**

- The seat belt and shoulder harness of each occupant of an aircraft should be properly fastened prior to takeoff and until the aircraft is completely stopped after landing. Seat belts should not be removed except when necessary activities require temporary removal, and they should not be removed below 1000 feet altitude without authorization of the pilot.
- 2. There should be no smoking during takeoffs, landings, or use of oxygen. Smoking is permitted during the flight only with the pilot's permission.
- 3. Passengers should keep clear of the controls and should not move around during the flight. If any maps or papers are used during the flight, they should be held securely so they do not interfere with operation of the aircraft. No object should be thrown from any aircraft in flight or on the ground.
- 4. At low altitudes, passengers can assist the pilot by keeping alert for hazards, particularly other aircraft, radio towers, and power and telephone lines. During landings the pilot may ask for assistance in seeing that the runway is clear of all aircraft or that there is tail rotor clearance.

# **B.6 Emergencies in Flight**

Passengers should be prepared for emergencies which may occur during a flight, particularly if the flight is over remote areas or water.

#### **B.6.1** Forced Landing

- 1. During a forced landing, passengers should follow the instructions of the pilot.
- The pilot may ask passengers to jettison doors, inflate flotation equipment, assist the injured, or exit the aircraft.
- 3. Passengers can also assist the pilot in activating emergency signaling equipment if requested.

#### **B.6.2** Water Survival

Certain safety and survival equipment is required in every aircraft operating over water on an extended flight. Safety and survival equipment includes a life vest or personal flotation device for every person and a wet suit if required by water and air temperatures.

#### **B.6.3 Emergency Locator Transmitters**

As standard safety equipment, aircraft have emergency locator transmitters (ELT) which are automatically activated in the event of a crash to send out a radio signal. The ELT has a normal range of 150 miles on a VHF frequency of 121.5 megahertz and a UHF frequency of 243.0 megahertz.

#### B.6.4 Helicopter Ditching Survival

- 1. Since relatively few helicopters are forced to ditch, there is limited information about the possible problems, and it is easy to underestimate the hazards involved in such an emergency landing.
- 2. Helicopter crews tend to believe that water provides a safe emergency landing surface and that ditching is a relatively simple maneuver. However, ditching is always a dangerous procedure, and helicopters have been lost in rivers and bays as well as in larger bodies of water. Unplanned ditchings have resulted from weather conditions, night operations over water, running out of fuel, and mechanical failure.
- 3. If ditching is anticipated, passengers should secure all tool boxes, cargo, and equipment that may be loose. They should remain securely strapped in their seats, locate the exits, and follow the directions of the pilot. Problems of escaping from an aircraft in the water include:
  - a. Inrushing water which tends to force cabin occupants into rear corners of cabin and to cause disorientation in locating exits.
  - b. Difficulty in locating personal flotation devices.
  - c. Difficulty reaching or opening exits. It is important to know where emergency exit releases are located prior to going down and to have doors positioned or latched to minimize amount of inrushing water.
  - d. Difficulty in getting to the surface because of dark or murky water.
  - e. Damage to aircraft or spilled fuel.

#### **B.6.5** Accident Reporting

- 1. In case of an accident, the appropriate agency should be notified.
- 2. An aircraft accident form should be completed and expedited to the proper authorities.

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